THE RECOVERY OF VALUABLE VAPOUR PHASE SOLVENTS USING ACTIVATED CHARCOAL CLOTH

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TO

MUM, DAD AND KATH

FOR ALL THEIR ENCOURAGEMENT AND PATIENCE

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SUMMARY

THE RECOVERY OF VALUABLE VAPOUR PHASE SOLVENTS USING ACTIVATED CHARCOAL CLOTH

The removal of toluene and perchloroethylene vapours from air by adsorption onto activated charcoal cloth and their subsequent recovery by regenerating the cloth with low pressure steam was investigated. Additionally, present market areas for activated charcoals have been presented as well as outlining new market fields for activated charcoal cloth.

The potential of charcoal cloth for use on solvent recovery plants has been assessed using both batch and continuous equipment. The charcoal cloth which on average measured 0.72m by 0.60m and weighed 0.045Kg was wound on a former and placed in a glass column for the batch operations. The experimental results in general could be represented by Langmuir isotherms and the maximum adsorbate-adsorbent mass ratios obtained were, 0.830 for perchloroethylene and 0.388 in the case of toluene. An approximate working charge of 52% corresponding to a steam-solvent mass ratio of 2.5 was measured for both solvents. Cyclic efficiencies were evaluated at 45.8% and 24.2% for the chlorinated solvent and toluene respectively.

Empirical equations used to describe the equilibrium relationships and the adsorption waves were computed and are presented.

The experimental apparatus constructed for the continuous operations basically consisted of four sections; an air solvent mixing region, the adsorber, regenerator and solvent recovery unit. A continuous length of cloth was transported over a series of rollers through a 1m length of duct counter currently to the flow of solvent vapour-air mixture. The cloth was then passed through the regenerator where solvent was desorbed by steam.

Measurements of the cloth's residual activity showed an immediate drop after one regeneration, but then remained fairly constant with increasing number of regenerations.

Evidence in this thesis exists to show that at present activated charcoal cloth would be uneconomical for the batchwise recovery of solvents and more development work has to be carried out before the cloth can be established as an effective material for the continuous recovery of solvent vapours. However, it is demonstrated that charcoal cloth can be beneficial in numerous other areas.

Keywords

Charcoal Cloth, Vapour Phase Adsorption, Batch Operations, Continuous Operations, Steam Regeneration, Markets.

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CHAPTER 1

INTRODUCTION

1 INTRODUCTION

The characteristics of powdered and granular forms of active carbons are well established, but the knowledge of activated charcoal cloth properties, an alternative form of activated carbon manufactured since the early 1960s, is limited. One such area is the ability of activated charcoal cloth in the dynamic recovery of valuable vapour phase solvents from air on an industrial scale.

This thesis begins by contrasting some of the properties of granular carbons and activated charcoal cloth and includes results of experiments undertaken at CDE Porton Down which investigated the dynamic uptake of solvent vapours by these two carbon forms. A general survey of adsorption theory is then presented followed by a discussion of factors affecting adsorption. The techniques available for recovering the adsorbate, thus regenerating the adsorbent, are outlined.

The performance of charcoal cloth in adsorbing perchloroethylene and toluene solvent vapours from air under dynamic conditions has been studied. This was carried out in batch equipment and in a continuous rig and the experimental details of both are described. Low pressure steam was used to desorb the organic solvent in question from the cloth and measurements of the material recovered were taken. The effect of the number of regenerations upon the activity of charcoal cloth is also reported.

Mathematical models have been developed for the batch rig, whereby relationships have been drawn up to express the equilibrium data and predict the adsorption wave.

Finally, a Chapter has been included to highlight possible market areas in which the characteristics of activated charcoal cloth could be exploited.

CHAPTER 2

LITERATURE SURVEY

2 LITERATURE SURVEY

2.1 Brief History

Throughout history carbon has been known as an adsorbent. As long ago as 1550 BC charcoal was used in medicine and wood chars were used to treat some ailments in the time of Hippocrates[1,2]. In the thirteenth century sugar solutions were purified using carbon materials. Scheele[2], in 1773 discovered that gases were adsorbed onto carbon and the decolourizing properties of activated charcoal were realized by Lowitz[3] some twelve years later. Early in the nineteenth century, powdered bone char was used as a purifying agent in the sugar industry. Bone char was initially discarded when it became saturated, but later regenerated when the material became scarce.

In 1865 coconut char was found to have gas adsorbing powers[2] and it was about the same time Stenhouse[4] produced a decolourizing carbon by heating a mixture of tar, flour and magnesium carbonate. A carbon with decolourizing properties, some 20-50 times greater than bone char, was developed by Bussy[5] in 1882 by heating blood with potash. The development of more modern commercial activated carbons stems from work undertaken by Ostrejko[2] at the beginning of the century. It involved selective oxidation of carbonaceous materials using carbon dioxide at elevated temperatures, and in another process such materials were treated with metallic chlorides.

In the middle of the twentieth century activated charcoal proved popular for the recovery of benzol from coal gas and the removal of impurities such as hydrogen sulphide and some hydrocarbons from natural gas[2].

More recently, since 1970, Maggs[6,7] has developed a manufacturing process which produces a highly adsorptive charcoal in the form of a cloth.

2.2 Manufacture of Activated Carbons

This Section includes a general discussion on some of the processes used in manufacturing activated carbons and pays particular attention to the process used to produce charcoal cloth.

2.2.1 General

Activated carbon is made up of several substances, none of which can be independently identified by chemical analysis or said to have a definite structural formula[8].

A list of some of the main base materials which have been investigated for producing activated carbon is shown below.

Beat Sugar Sludges
Blood
Bones
Carbohydrates
Coal
Coconut Shells
Coffee Beans
Fish
Fruit Pits
Graphite
Kelp and Seaweed
Lampblack
Lignite
Molasses
Nut Shells
Oil Shale
Peat
Petroleum Acid Sludge
Rubber Waste
Wood

Table 2.1 - Source materials studied for the production of activated carbon

Published work of Bancroft[9] and Courouleau[10] outlines some of the methods used to produce activated carbon. Each of the carbonaceous materials undergoes carbonization and activation. Carbonization is normally carried out by heating the source material to temperatures below 600°C in the absence of air and the effectiveness of this process is improved by impregnating the base material with metallic chlorides. Activation, which increases the adsorbability of the char produced by the carbonization process involves oxidation of the char between 800° and 900°C using steam or carbon dioxide.

2.2.2 Commercial Powdered and Granular Activated Carbons

There are a few processes in commercial operation today used to produce powdered and granular charcoal. As outlined in Section 2.1.1, the general scheme for producing activated charcoals involves the two-stage process of carbonization followed by activation, the operations normally being carried out in multiple-hearth furnaces, rotary kilns or batch furnaces[12]. The powdered forms of activated carbon are usually ground to give a distribution of particle sizes in the range 5-100μm.

Powdered and granular charcoals are produced simultaneously by a process known as fluidized bed steam activation[13]. A flow diagram of the process is shown in Figure 2.1. Superheated steam is used to fluidize a bed of anthracite particles which are heated up to 950°C by gas burners. Powdered carbon is removed from the top of the reactor, cooled, then crushed to its desired specification. Granular charcoal leaves the base of the reactor and after cooling is screened into its various size fractions.

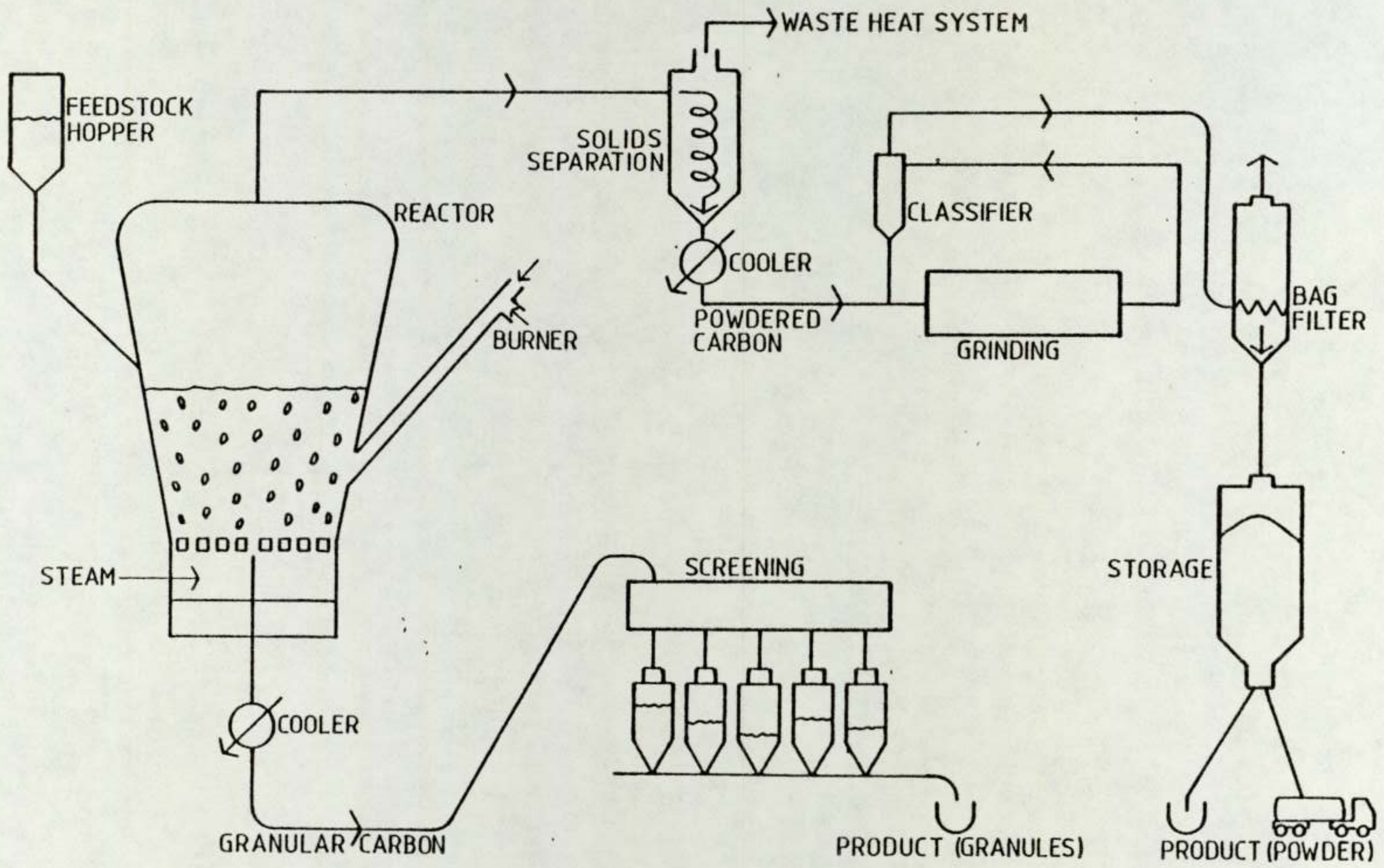


FIGURE 2.1 - A PROCESS FOR THE PRODUCTION OF GRANULAR AND POWDERED CHARCOALS

2.2.3 Carbon Cloth and Fabrics

Carbon cloth is made by spraying a mixture of ground granular charcoal and latex onto a non-woven cloth such as viscose nylon and terylene[14].

Activated carbon in fibrous form has been produced by spinning a synthetic fibre impregnated with activated carbon[15].

2.2.4 Charcoal Cloth

Viscose rayon cloths are the raw materials used in the manufacture of activated charcoal cloth[16]. The process for producing the cloth was conceived and developed by Maggs at CDE Porton Down. The method of manufacture is now described and a diagram of the process is shown in Figure 2.2.

2.2.4.1 Pre-treatment

Pre-treatment of viscose rayon cloth essentially comprises of three stages: impregnation, drying and flexing[17,18,19]. The yield and breaking strength of the carbonized product are increased by spraying raw material with a mixture of zinc chloride, aluminium chloride and ammonium chloride. Heat treatment during carbonization is also made more flexible. The impregnated cloth is then dried by a stream of warm air and infra-red heaters. Finally, before carbonization, a spiral roller breaker is used to flex the cloth. This is undertaken to prevent the formation of a brittle carbonized product and reduce the variation in the cloth's breaking strength.

2.2.4.2 Carbonization and Activation

At CDE Nancekuke, the pre-treated viscose rayon cloth was carbonized in one furnace before being activated in a second furnace[20]. The production

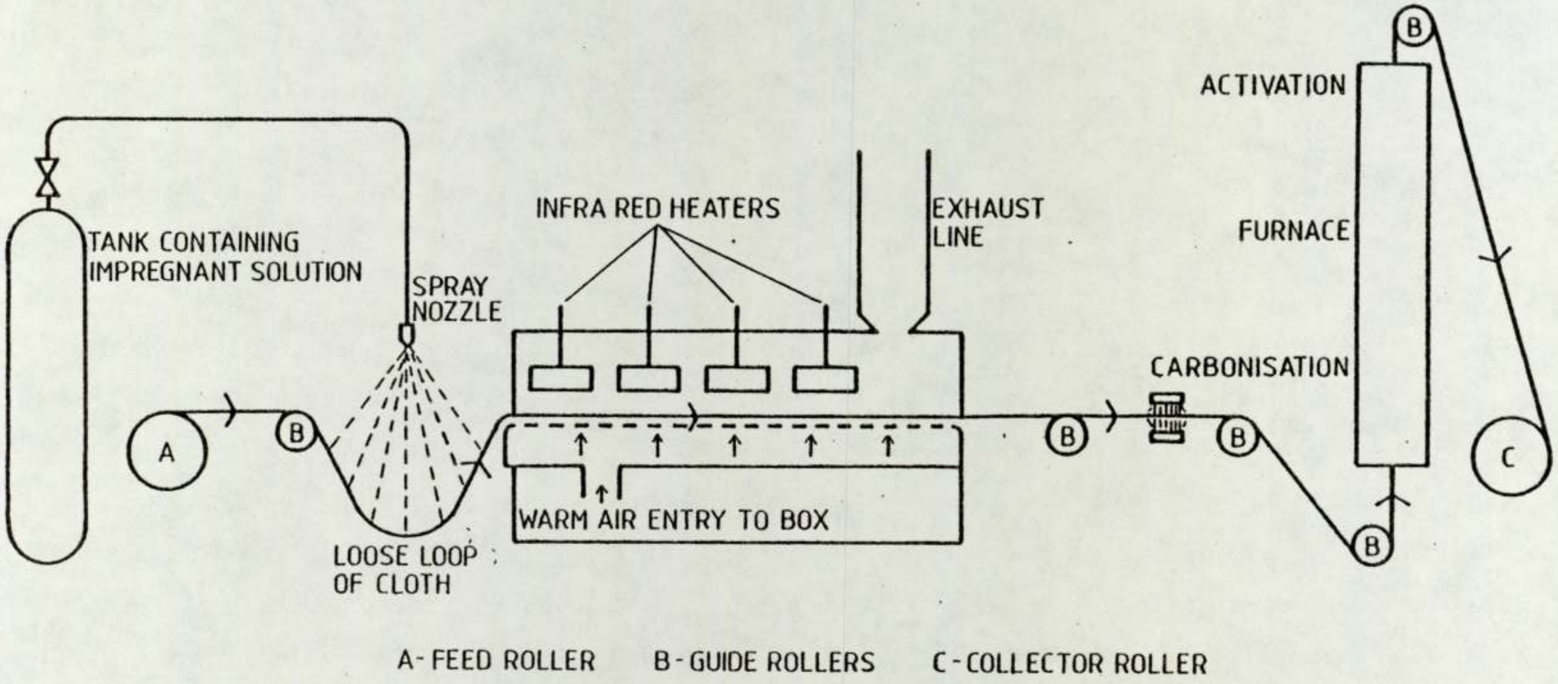


FIGURE 2.2 - PROCESS FOR THE MANUFACTURE OF ACTIVATED CHARCOAL CLOTH

of charcoal cloth, now carried out at CDE Porton Down combines the carbonization and activation processes into one furnace[21]. The pre-treated cloth is drawn through the electrically heated furnace by a constant speed roller mechanism. Carbonization of the cloth is achieved in the lower part of the furnace in a nitrogen atmosphere at about 275°C. The activation process is carried out in the upper section of the furnace at temperatures in excess of 800°C whereby steam or carbon dioxide reacts at the internal surface of the char to produce a highly adsorptive charcoal[7].

2.3 Properties of Activated Carbons

This Section describes and compares the properties of granular and powdered charcoals, carbon cloths and fabrics with activated charcoal cloth.

2.3.1 Effect of Raw Material Properties and Manufacturing Methods

The properties of activated charcoals are influenced by the char properties which, in turn, depend on the characteristics of the raw material and on the manufacturing conditions. These effects are now described.

2.3.1.1 Raw Material Properties

Almost any substance which is essentially composed of carbon can be activated; the more common base materials having been included in Section 2.1.1. However, the material must be hard so as to resist attrition when in use and low in volatile material for a high product yield[13]. The pore size of the carbon can be varied by using different raw materials. Coconut shells, for example, yield a granular charcoal with small pores[22].

A variety of textile cloths and yarns have been examined at CDE Porton Down[6,20]. The results infer that, after carbonization, a high yield of consistently strong cloth was produced from rayon cloth (Reference No 4325) as supplied by Rosebank Weaving Company Limited, Belfast.

However, viscose rayon cloth can be obtained in a number of weaves from the same supplier. There are also variations in filament properties constituting a thread. The influence of such factors on the properties of activated charcoal cloth have yet to be investigated[23].

2.3.1.2 Manufacturing Conditions

The production of activated charcoals, as previously outlined essentially involves pre-treatment carbonization and activation of the raw material. The grading of powdered and granular carbons into various particle size fractions is also carried out. Determination of the particle size depends on the desired adsorption effect and permissible pressure loss[22].

The efficiency of the adsorption process and the capacity of the adsorbate carbon for certain gases can be increased by impregnating the carbon with an inorganic compound that will react with the material which is to be adsorbed. Interaction of the base cellulose material with Lewis acids has been found to increase the yield and strength of the carbonized cloth[6].

For a particular raw material, the effectiveness of carbonization depends on the maximum carbonizing temperature, the flow rate of inert gas and throughput of the source material. Studies regarding the optimum operating conditions for producing a char from viscose rayon cloth have been undertaken by Morgan and Ramsay[20].

The degree of activation which determines the internal surface area of a charcoal greatly influences its adsorptive properties[22]. During the early stages of activation many pores are too small to admit large molecules, but as activation proceeds, the pores are enlarged therefore allowing a greater number of larger molecules to be adsorbed[24,25]. Increasing activation increases both the flexibility and electrical resistance of charcoal cloth. In the process whereby activation follows carbonization in the same furnace, the breaking strength of the activates rises to a maximum with increasing activation. However, an immediate decrease in the breaking strength of the charcoal cloth is apparent on successive activation when the char is cooled and handled between these two stages[26].

2.3.2 Physical Properties of Activated Charcoals

Activated charcoals or carbons contain a complex network of pores of various shapes and sizes, the distribution of which depends on the source material itself and the method and extent of activation.

Powdered activated charcoal is a very fine material and can create a dusty working environment. Because of this it is invariably handled in slurry form. Granular or nutshell charcoals, although not dusty materials, do undergo abrasion and degradation, and, as for powdered charcoals, are handled as a slurry. Pneumatic handling is effective in transporting powdered and granular charcoals, but is costly[13]. However, charcoal cloth, carbon cloth and fibres impregnated with activated carbon can be handled inexpensively and with relative ease.

Impregnated charcoal fabrics can be made of up to 80% (by weight) of charcoal[15]. Arons et al[27], however, have suggested that polymers loaded with activated carbon are not entirely satisfactory. Since carbon particles are physically entrapped in the polymeric binders, the sorption efficiency of the charcoal is reduced. More carbon must be used to compensate, thus increasing the weight of the material and decreasing air permeability.

Carbon cloth, produced quickly and inexpensively, usually contains 4 x 10^{-5}Kg (40mg) of carbon per square metre of cloth. However, carbon cloth has two main drawbacks. The first is that the latex reduces the carbon's adsorptive capacity by blocking pores within the carbon. Secondly, carbon cloth cannot be regenerated since temperatures necessary for effective desorption melt the latex thus causing the carbon to become detached from the cloth.

Charcoal cloth is composed of 100% activated charcoal fibres in the form of a woven cloth. It is a strong flexible medium, resists shock and vibration and needs neither container nor cannister[25,28]. The physical properties of charcoal cloth are summarized in Table 2.1[25,29].

Cloth weight	0.11 kg/m^2
Bulk density	1.90 - 2.80 kg/m^3
Surface density	0.08 - 0.14 kg/m^2
Specific surface area (BET N_2 method)	>1.3 x 10^6 m^2/kg
Specific shadow area	90 - 92%
Air-flow resistance	14m H_2O/m/s/layer
Thermal conductivity	0.08 watts/m/k
Electrical conductivity	20 - 50 ohm/square
Heat of wetting	0.063 J/kg (benzene)
	0.025 J/kg (in silicone)
Breaking strength	340 - 170 kg/m (warp)
	189 - 174 kg/m (weft)

Table 2.2 - Properties of Charcoal Cloth

The bulk density and specific surface area of powdered and granular charcoals varies with the size fraction of the charcoal. These properties are compared with those of charcoal cloth in Table 2.3[13,22,25,29].

	Powdered Charcoal	Granular Charcoal	Charcoal Cloth
Bulk Density	4900-7100 kg/m^3	6100 kg/m^3	1.90-2.80 kg/m^3
Specific Surface Area (BET N_2 Method)	4 x 10^6 - 11 x 10^6 m^2/kg	6 x 10^6 - 12 x 10^6 m^2/kg	>13 x 10^6 m^2/kg

Table 2.3 - Comparison of bulk density and specific surface area of powdered and granular charcoals and charcoal cloth

The Table clearly shows that in comparing granular and powdered carbons with charcoal cloth, the latter has a much lower bulk density. The adsorptive capacity of charcoal cloth per unit weight is, in general, greater than granular or powdered charcoal, since it has a higher specific surface area. Charcoal cloth can be folded, pleated and laminated to a variety of fabrics without deterioration or loss in adsorptive capacity[16,27,28].

2.3.3 Adsorptive Properties of Activated Charcoals

The adsorptive properties of activated charcoals are discussed in three Sections: Liquid Applications, Vapour Phase Applications and Potential Controlled Adsorption and Desorption with regard to Charcoal Cloth.

2.3.3.1 Liquid Applications

Activated charcoals have a variety of liquid applications and these are outlined in Chapter 7. By adsorbing pollutant molecules, the active charcoals are used for decolouring and removing undesirable tastes and odours from liquids[30].

Since powdered or pulverized charcoals have a relatively low initial cost, they are used in batch operations whereby the liquid to be treated is mixed with the charcoal and then separated by filtration. Following this, the powdered carbon is discarded[2,12,13]. Dosages are generally only a few parts per million because rapid contact of the pollutant molecules is brought about by the numerous fine charcoal particles which scatter throughout the liquid[31].

Liquid to be purified in a granular charcoal system is passed continually through the carbon bed. Granular charcoals are usually regenerated for further use and thus offsets the comparatively high initial cost. Such charcoals are also advantageous in that they adsorb more impurity than can be accomplished by powdered charcoals in batch operations[30].

However, Wilkins[32], found that under normal conditions the capacity of charcoal cloth with regard to the adsorption of phenol and 2-chlorophenol from water was 90% and 98% respectively, some three to four times greater than for granular charcoal. The pollutants were burnt off and the cloth regenerated by passing the material through a heater using continuous apparatus.

2.3.3.2 Vapour Phase Applications

In vapour phase adsorption processes activated charcoals are mainly used for solvent recovery and purification of gases and air[13]. Granular charcoal is commonly used in vapour phase applications and adsorption usually takes place under dynamic conditions.

Work undertaken at CDE Porton Down has dealt with the dynamic adsorption of certain solvents onto charcoal cloth and granular carbon[33,34,35,36,37]. The uptake of solvent per unit mass of charcoal and adsorption rates have been compared under dry conditions and at 80% relative humidity. Granular charcoal (Reference LS 796, 14-25 BSS ex Sutcliffe Speakman and Company Limited) and charcoal cloth (Reference NKE 123W) were used. A list of the solvents studied and the experimental conditions for dynamic adsorption are shown in Table 2.4.

	Bed Depth			
Solvent	Charcoal Cloth (layers)	Granular Charcoal (m)	Flow Rates (m^3/s) ($\times 10^4$)	Concentration (kg/m^3) ($\times 10^3$)
Chlorobenzene	10-40	0.01-0.04	0.166,0.250,0.332	6.0,9.0
Chloroform	10-40	0.01-0.04	0.166,0.332	5.5,11.0
Trichloroethylene	10-40	0.01-0.04	0.166,0.332	4.5,9.0
Carbon Tetrachloride	10-40	0.01-0.04	0.166,0.332	4.0,8.0
Penthrane	10-40	0.01-0.04	0.166,0.332	10.0,20.0

Table 2.4 - Experimental conditions for dynamic adsorption of solvent vapours on activated charcoal cloth and granular activated charcoal

A comparison of the average percentage weight uptakes and rates of adsorption for charcoal cloth and granular charcoal under the conditions quoted in Table 2.4 are summarized below in Table 2.5. The results were obtained for a penetration end point whereby the effluent concentration had reached one two-hundredth of the inlet composition.

Solvent	Conditions	Charcoal Type	Average % Weight Uptake (%)	Average Adsorption Rate (kg mols/s)
Chlorobenzene (C_6H_5Cl)	Dry	Charcoal Cloth	58.2	9.14×10^{-8}
		Granular Charcoal	34.8	9.14×10^{-8}
	80% RH	Charcoal Cloth	44.7	8.73×10^{-8}
		Granular Charcoal	16.0	11.79×10^{-8}
Chloroform ($CHCl_3$)	Dry	Charcoal Cloth	46.7	8.11×10^{-8}
		Granular Charcoal	27.9	8.13×10^{-8}
	80% RH	Charcoal Cloth	8.5	9.29×10^{-8}
		Granular Charcoal	2.4	8.62×10^{-8}
Trichloroethylene (C_2HCl_3)	Dry	Charcoal Cloth	52.1	6.10×10^{-8}
		Granular Charcoal	30.5	7.00×10^{-8}
	80% RH	Charcoal Cloth	18.6	6.27×10^{-8}
		Granular Charcoal	4.7	5.98×10^{-8}
Carbon Tetrachloride (CCl_4)	Dry	Charcoal Cloth	61.0	4.60×10^{-8}
		Granular Charcoal	29.5	5.32×10^{-8}
	80% RH	Charcoal Cloth	15.6	4.73×10^{-8}
		Granular Charcoal	1.7	4.43×10^{-8}
Penthrane ($CHCl_2.CF_2.OCH_3$)	Dry	Charcoal Cloth	56.1	11.07×10^{-8}
		Granular Charcoal	33.3	10.58×10^{-8}
	80% RH	Charcoal Cloth	32.5	10.28×10^{-8}
		Granular Charcoal	7.2	10.64×10^{-8}

Table 2.5[33-37] Average percentage weight uptakes and adsorption rates for the dynamic adsorption of particular solvents on charcoal cloth and granular charcoal under dry conditions and at 80% relative humidity

It is shown in Table 2.5 that for all solvents, the charcoal cloth adsorbed more vapour per unit weight than did granular charcoal. The capacity of both charcoals is reduced under humid conditions, but there is a much greater deterioration in the granular charcoal. For a particular solvent the adsorption rate in terms of the number of moles adsorbed in unit time is similar for both charcoals under dry conditions and at 80% relative humidity.

Experiments at Porton Down investigating dynamic adsorption of vapours onto activated charcoal cloth have only involved relatively small quantities of material. This research project, however, looks at the dynamic adsorption of solvent vapours onto larger quantities of charcoal cloth which are incorporated into process systems.

2.3.3.3 Potential Controlled Adsorption and Desorption Using Activated Charcoal Cloth

Although porous carbons possess a large surface area available for adsorption, they are not normally electrically conductive because of their amorphous structure. Graphite, however, does conduct electricity, but has a low surface/volume ratio. Charcoal cloth has a high adsorptive area and is electrically conductive because of its graphitic structure[38]. It also presents a convenient physical form as an electrode which can be used in the removal of certain substances from a flowing solution.

Evidence from preliminary experiments involving organic polar compounds including aniline and phenol, suggests that the amount of polar material adsorbed by and desorbed from activated charcoal cloth can be controlled by electrical means[38,39].

2.4 Recovery of Solvents from Gaseous Effluents Using Activated Charcoal

A wide variety of volatile organic solvents are involved in the production of numerous products. For example, toluene, benzene and xylene are used as ink thinners in the printing industry and acetone is employed in the manufacture of rayon[40]. Also, perchloroethylene is used as the major cleansing agent in the dry cleaning industry. One consequence of such operations is that large quantities of volatile organic vapours may be emitted to the atmosphere. Not only may stricter legislation be implemented regarding toxic emissions, but also the rising costs of volatile organic materials can render an economical solvent recovery process.

Section 2.4.1 outlines some of the processes involved in controlling toxic emissions and a discussion of the recovery of solvent vapours using activated charcoal is included in Section 2.4.2.

2.4.1 Controlling Toxic Emissions

Several methods have been used to control emissions of organic solvent vapours[41].

The concentration of the polluting solvent in the air can be reduced by removing the pollutants from a particular process in a ventilating air stream and/or dispersing the effluent from tall stacks. Obnoxious, but non-toxic odours are nullified by masking with perfume, but this effectively further 'pollutes' the air.

Condensers are useful for reducing high concentrations of solvent in air, but the discharge levels are limited by the cooling water temperature. Better recovery can be achieved using refrigerated condensers, but water must not be present to foul the condenser with ice.

The use of scrubbers whereby non-volatile organics are used to remove the volatile organics from the carrier gas have technical and economic limitations, the reason being that the spent scrubbing fluid has to be re-used without removing adsorbed organics. Additionally, desorption is expensive and the concentration of the adsorbed organics in the scrubbing fluid must be low for the process to be effective.

Fume incineration is an effective way of controlling toxic emissions, even in low concentrations. However, it is an expensive recovery method since the valuable pollutants are destroyed.

In the recovery of solvents using activated carbons, the polluting organics are adsorbed from an exhaust gas stream onto the surface of the charcoal. The activated charcoal is reactivated using steam which desorbs the pollutant molecules. The steam-solvent vapour mixture is condensed before solvent is recovered. This method of controlling toxic emissions is effective and can be very economical.

2.4.2 Recovery of Valuable Vapour Phase Solvents Using Activated Charcoal

2.4.2.1 Pre-treatment

The solvents which are to be recovered are usually carried away in an air stream. This minimizes the leakage of solvent vapour into a working environment from process equipment enclosures and prevents the formation of explosive mixtures[42].

In order to maintain a low pressure drop across an activated charcoal bed and maximise its adsorptive efficiency, any particulate matter entrained in the air-solvent stream is removed by filtration or an electrostatic precipitator. The solvent laden air is normally then dried and cooled to temperatures below 311K (100^0F) before being fed to the adsorber[43,44].

2.4.2.2 Adsorption-Desorption Cycles

A typical solvent recovery plant using activated charcoal is shown in Figure 2.3. The solvent laden air is blown into a vessel containing the activated charcoal which adsorbs the solvent vapour and allows the stripped air to pass through[45]. These vessels are known as adsorbers and there are usually two or more on a solvent recovery plant for continuous operation. The solvent air mixture is transferred to another adsorber when some predetermined solvent concentration is reached in the effluent air.

When solvent from a solvent laden air stream is first adsorbed on pure activated charcoal, a large initial quantity of solvent is adsorbed. The charcoal is regenerated using low pressure steam when a proportion of the solvent is desorbed. The charcoal, left hot and wet by the steam, is cooled and dried by blowing air into the adsorber. When further solvent laden air is passed through the regenerated charcoal, more solvent laden air is adsorbed up to the initial saturation point. The difference between the initial quantity of solvent adsorbed and the amount remaining on the carbon after desorption is known as the cyclic adsorption figure. Typical initial and cyclic adsorption values for a variety of solvents onto granular charcoal are shown in Table 2.6[46,47].

A variety of solvent recovery plant designs have been developed over the years. Rowson[48] found that recovery efficiency of carbon disulphide is similar for fixed and fluidized beds of activated carbon, but steam consumption is slightly lower in the latter case. Solvent recovery processes have been reviewed by Avery and Boiston[49] who stated that, in general, a moving bed is preferable since more charcoal surface area is available for mass transfer. However, problems do arise with granular charcoal due to attrition and transport losses.

Both horizontal and vertical adsorbers have been designed and recently

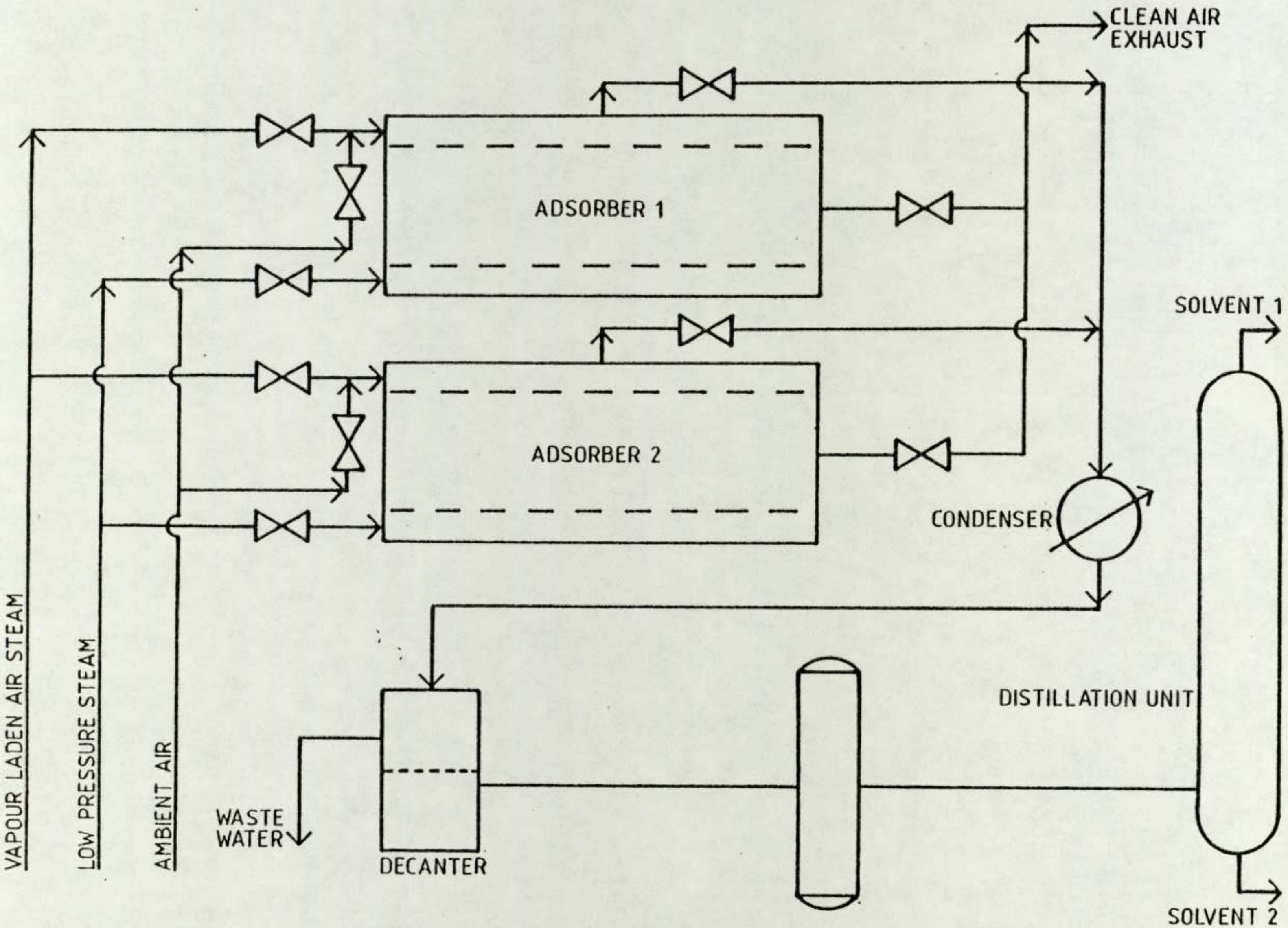


FIGURE 2.3 - TYPICAL SOLVENT RECOVERY PLANT

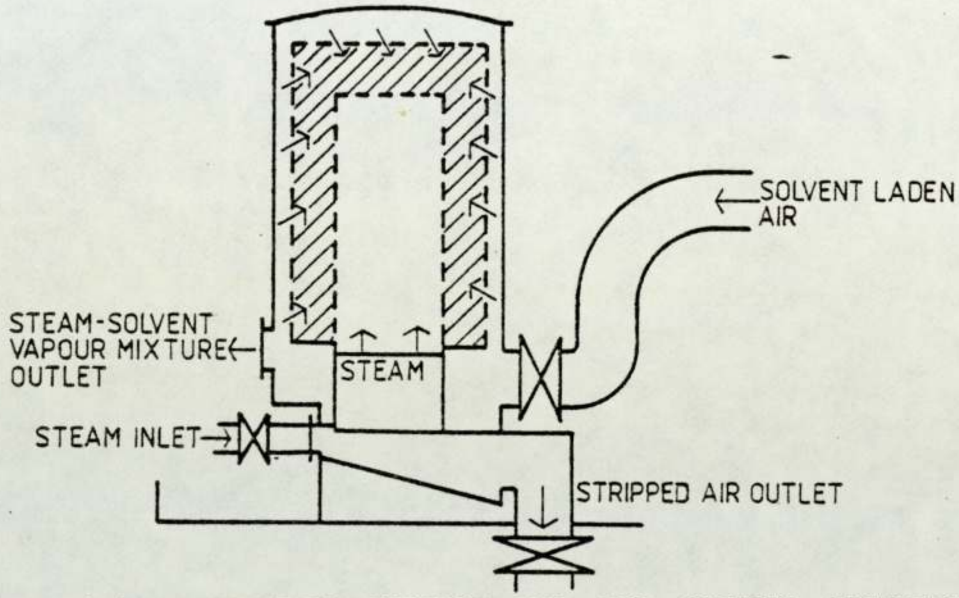


FIGURE 2.4 - VERTICAL ANNULAR BED FOR GASEOUS ADSORPTION WITH ACTIVATED CHARCOAL

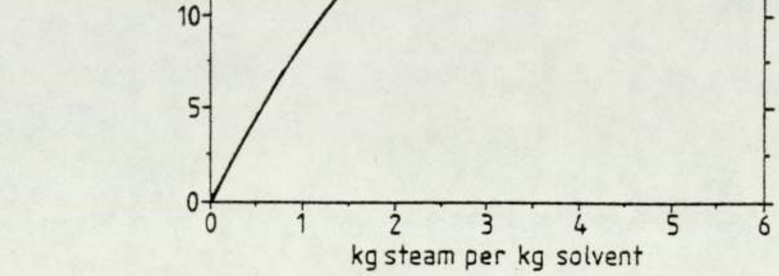


FIGURE 2.5 - A PLOT OF WORKING CHARGE AGAINST STEAM REQUIREMENTS FOR ACETONE RECOVERY

Solvent	Concentration (vol %)	Adsorption (wt %)		+Steam Ratio
		Initial*	Cyclic	
Methylene Chloride	1.00	28.3	17.3	1.4
Arklone P	0.50	44.9	20.8	1.4
Acetone	1.00	20.3	12.5	2.3
Toluene	0.40	23.3	9.6	3.5
n Heptane	0.12	22.4	5.9	4.3
Trichloroethylene	0.50	44.6	19.9	1.8
Hexane	0.48	21.3	8.2	3.5

* Initial adsorption refers to the solvent adsorbed by virgin carbon when the solvent concentration in the air stream is of the order 20ppm

+ Steam ratio refers to the total steam used in desorption and is inclusive of condensate and moisture condensed on the carbon bed

Table 2.6 - Some typical results of solvent adsorption by granular charcoals in solvent recovery practice

Hill[47,48] suggests that vertical annular bed designs have several advantages. One drawback of horizontal beds is that condensate falls back onto the carbon bed and this is minimized in a vertical annular adsorber. Good uniformity of air flow, emptying and filling the adsorber with new carbon being gravity assisted and using less floor space than horizontal vessels are also beneficial. The annular vertical arrangement also reduces the load on the main condenser since the cooler outer shell of the adsorber acts as an air condenser. A vertical annular adsorber used for gaseous adsorption with activated carbon is shown in Figure 2.4.

Bearing these factors in mind, solvent recovery on a fixed bed of activated charcoal cloth is carried out using a vertical arrangement. A full description of the equipment used is included in Chapter 4.

2.4.2.3 Solvent Recovery

The steam-solvent vapour mixture discharged from the adsorber is passed to a shell-and-tube-type heat exchanger where it is condensed. Treybal[45], and more recently Drew[50], have outlined several examples of solvent recovery processes.

If the solvent is immiscible with water, separation is achieved by simply decanting, but the recovery of the solvent from a miscible mixture is usually carried out in a distillation column. The use of a continuous decanter, continuous distillation and batch distillation columns may be necessary if the condensate is a mixture of both water-miscible and -immiscible solvents.

2.4.2.4 Corrosion

Published work of Drew[50] also discusses the influence of the types of solvent which are to be recovered on the materials of construction for the plant equipment.

Carbon steel equipment can usually be used when distilling wet or dry organic solvents, but serious corrosion does arise if both organic and inorganic acids and salts are present. The use of carbon steel as a construction material should also be avoided if halogenated solvents and esters are to be recovered because they hydrolyse during steam regeneration forming corrosive acids. Small volumes of acid resulting from the decomposition of ketones in contact with active carbon have also been known to corrode solvent recovery plant units.

Therefore corrosion resistant materials must be used in the recovery of some solvents. Stainless steel is often selected when esters are handled but high nickel alloy vessels or glass-lined equipment is installed if mineral acids formed from halogenated solvents are present.

2.4.2.5 Safety

Again both Treybal[45] and Drew[50] have discussed the safety aspects associated with solvent recovery plants using activated carbon.

Since most solvents are flammable and form explosive mixtures with air, it is essential to maintain solvent concentrations well below their explosive limits. Solvent recovery plants must, therefore, incorporate continuous combustible gas analysers which shut down the process or sound an alarm if the solvent concentration reaches some pre-determined value. In recent literature Hill[46,47] has suggested that single beam/two wavelength infra-red gas analysers are sensitive and very satisfactory. Gas analysers are also involved in switching the adsorber from an adsorb mode to a desorb mode by continuously measuring the effluent solvent concentration from an adsorber.

The adsorption of solvent vapours onto activated charcoal is generally carried out at atmospheric pressure. However, since steam for regeneration and air for cooling the charcoal are introduced at elevated pressures, the equipment could rise above design pressures in the event of a blockage or instrument failure. Relief devices must therefore be used.

A temperature sensor is normally positioned at the outlet from the condenser to protect against cooling water failure. In the case of an undue temperature rise an alarm is generally sounded and the steam is shut down.

2.4.2.6 Economics of Solvent Recovery Plants Using Activated Carbon

The economic aspects of solvent recovery plants using activated carbon have recently been discussed by Young[51] who divides the cost into capital expenditure and operating expenditure.

2.4.2.6.1 Capital Expenditure

Several factors contribute to the capital cost of a solvent recovery system using activated carbon.

The air flow-rate through an adsorber vessel should not exceed 0.5 m/s if a high adsorption efficiency is to be maintained. This figure influences the cross-sectional area of charcoal available to the air which, in turn, determines the number of adsorbers to be used.

Generally, the higher the solvent concentration in the air, the more active carbon is required. If there are a number of constituent organics in the air stream which are to be recovered, then the cost of auxiliary equipment must be met. As outlined in Section 2.4.2.4, the type of solvents entrained in the air stream determines the construction material for the plant equipment. For example, if chlorinated solvents are to be recovered, a copper plant would prove more economical than one of plastic coated mild steel since the former has a much longer plant life.

The civil work to install a plant can demand a large percentage of the capital cost. They are often constructed in factories with limited space and in some cases such plants have been mounted on the roof of factories. Ducting costs for air-solvent mixtures can be high, so it is important to minimize the distance of the plant from the machinery.

A range of gas analyser controls with varying degrees of sophistication has been developed for solvent recovery plants. The capital cost of a plant is increased by fitting elaborate gas analysers, but savings are made over more conventional time-elapsed systems from a more effective control of steam.

2.4.2.6.2 Operating Expenditure

The running costs for a solvent recovery system using activated carbon can be separated into four main areas: steam usage, electricity, cooling water and maintenance and labour requirements.

Steam is an expensive commodity and any savings would therefore be favourable. As shown in Table 2.6, the amount of steam required is determined by the organic solvents being recovered. It also depends on the concentration of the solvent in the air stream and as stated above, gas analysers register the inlet solvent concentration and adjust the steam rate accordingly. The greater the volume of steam used, the more solvent is recovered. However, it is not economical to remove all the solvent in each cycle since the cost of more steam exceeds the value of the extra solvent recovered. For example, in Figure 2.5, Lee[52] shows that for acetone recovery, a steam-solvent ratio of about 2.5:1 is capable of removing 17% of the acetone adsorbed, this being known as the working charge. It is also shown that at high steam-solvent ratios there is only a small increase in the working charge. Steam costs are increased if the solvent is water soluble since a distillation column would be needed to effect separation.

The use of electricity on a solvent recovery plant stems from operating a blower used to pass the vapour through the activated carbon bed. The larger the volume of air to be handled, the higher the pressure drop across the bed, consequently the greater the electrical demand by the fan. For a particular air flow rate, the pressure drop also varies with the size fraction of the charcoal used. In comparison with the fan motor, electrical demand by the pumps which may be installed on the plant is negligible.

Cooling water is used to condense the steam-solvent vapour mixture discharged from an adsorber and may also be used if an air cooler is fitted to the plant. Cooling water is recirculated in order to minimize costs and water separated from the solvent can also be used as cooling water make-up.

The labour and maintenance cost of such plants are normally low. Since most solvent recovery systems using activated charcoal are generally automatic, Young[51] suggested that for medium-sized plants, four man-hours per 24 hour day would be adequate and also estimated a carbon usage of between 5 and 7 per cent. The presence or absence of materials other than solvent influences the degree of bed poisoning and to prevent build up of fines, Young recommended that the carbon be sieved at least every two years.

CHAPTER 3

GENERAL ADSORPTION THEORY

3 GENERAL ADSORPTION THEORY

Adsorption is a process whereby vapour and liquid molecules adhere to a solid surface as a result of intermolecular or chemical forces. It is a spontaneous, exothermic process and is accompanied by a reduction in the free energy of the system as well as a decrease in entropy. The degree of adsorption depends on the nature and character of the adsorbate (the component fluid which is removed from the carrier stream) -adsorbent (solid surface) system[53,54].

The adsorbent is made up of numerous fine pores, which vary greatly between adsorbents and generally have a high surface area per unit weight. For example, as outlined in Chapter 2, active carbons have surface areas between 1.00×10^5 and 1.20×10^6 m^2/kg in commercial adsorption processes.

Equilibrium and kinetic data are needed to design adsorption units and data for the life of the adsorbent is also required if the adsorptive system is a regenerative one. If equilibrium data is not available, it can be determined from theoretical isotherms which have been postulated[53], and the better known isotherms are described in this Chapter. The equilibrium distribution of adsorbate molecules between the adsorbent surface and vapour phase depends on a number of factors and their influence upon the effectiveness and efficiency of adsorption are discussed in Section 3.3. The general theory related to regeneration is included in Section 3.4 and finally, in Section 3.5, de-activation of adsorbents and its causes are outlined.

3.1 Adsorption Equilibria

3.1.1 Introduction

An adsorptive vapour system is said to attain equilibrium when the number of adsorbate molecules arriving at the solid adsorbent surface is equal to the number of molecules desorbing from the surface into the gaseous phase[55]. Numerous theories and equations have been put forward describing equilibrium between adsorbates and adsorbents, but no one theory has yet been devised which adequately explains the bulk of observations on a rigorous theoretical basis[53].

3.1.1.1 Single Component Adsorption

For a specific gas and unit weight of given adsorbent, the quantity of adsorbed vapour at equilibrium can be expressed as a function of the final pressure, p, and absolute temperature, T. Therefore,

$$W = f(p,T) \tag{3.1}$$

where W represents the quantity of gas adsorbed per unit weight of solid adsorbent. At constant temperature, the adsorption of a gas or vapour increases with increasing pressure. The adsorption isotherms for ammonia on charcoal carried out by Titoff[56] was amongst the earliest obtained and a diagram representing his experimental results is presented in Figure 3.1. It is noticed from these results that at both low adsorbate concentrations and high temperatures, Henry's Law can be applied,

$$V = cp \tag{3.2}$$

where V represents the volume of gas adsorbed and c is a constant.

Most experimental systems, however, can be fitted to Langmuir and

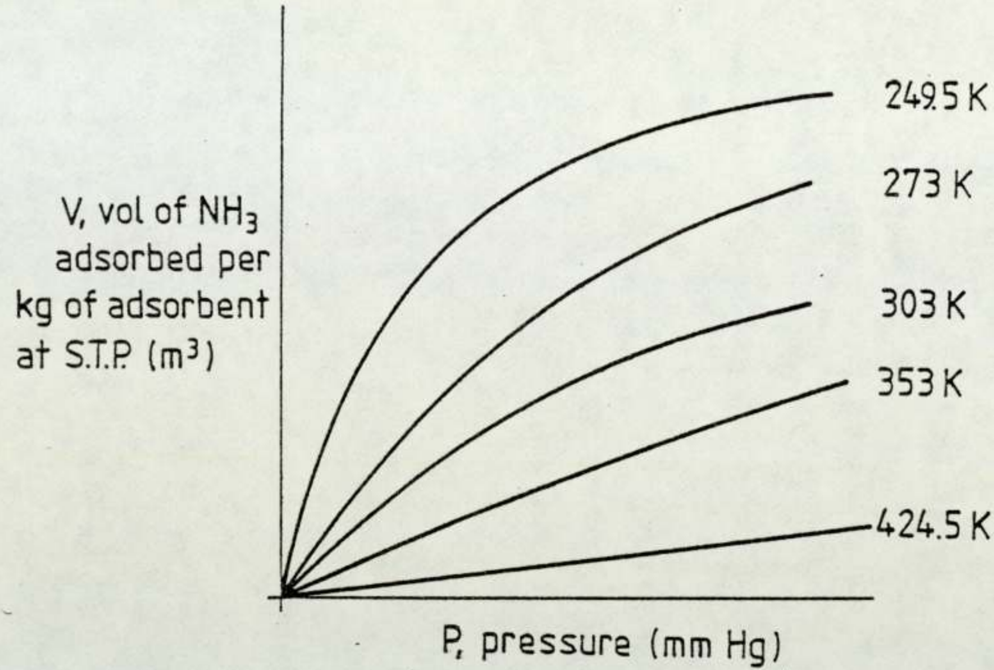


FIGURE 3.1 - ADSORPTION ISOTHERMS FOR THE UPTAKE OF AMMONIA ON CHARCOAL[56]

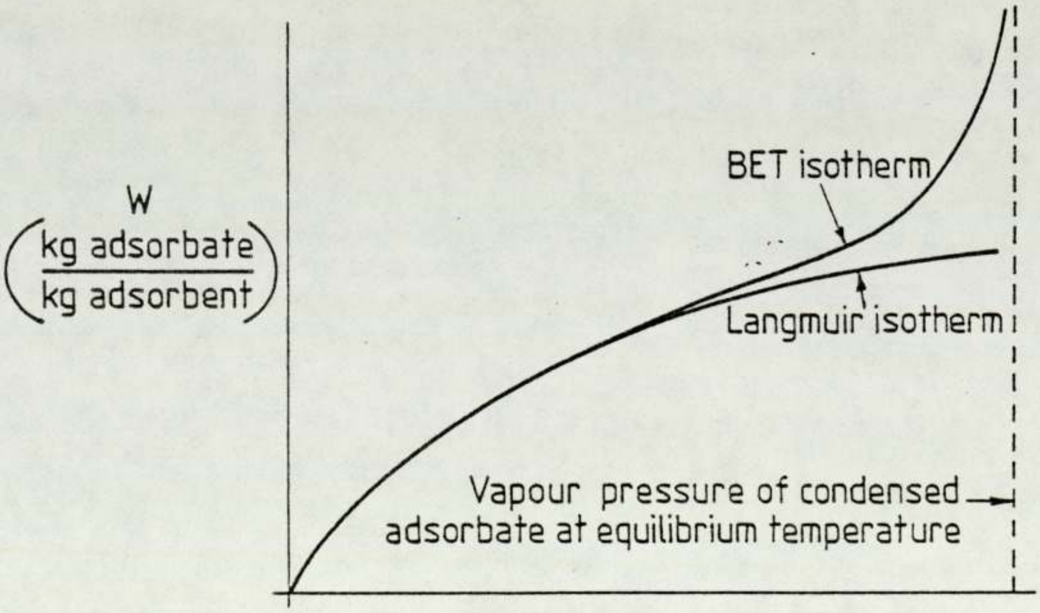


FIGURE 3.2 - LANGMUIR AND BET ADSORPTION ISOTHERMS[57,58]

Freundlich isotherms. One of the classic theories published has been put forward by Langmuir[57]. The theory assumes that the adsorbed layer is monomolecular and the force between atoms of the adsorbent surface and the adsorbate atoms rapidly decreases with distance between the atoms. A Langmuir isotherm is shown in Figure 3.2. Equation 3.3 represents an ideal adsorption isotherm as suggested by Langmuir,

$$\frac{V}{V_m} = \frac{kp}{(1 + kp)} \qquad 3.3$$

and may also be written in the form,

$$\frac{1}{V} = \frac{1}{kV_m}\left(\frac{1}{p}\right) + \frac{1}{V_m} \qquad 3.4$$

where k is a constant and V_m is the volume of gas adsorbed at STP per unit mass of adsorbent with a layer one molecule thick. Therefore, a plot of $1/_V$ against $1/_p$ should yield a straight line.

The empirical exponential equation of Freundlich[53] has subsequently been derived theoretically for low surface coverages and is written as,

$$V = k'p^{1/n} \qquad 3.5$$

where k' and n are constants. By taking the logarithm of both sides of Equation 3.5,

$$\ln.V = \ln.k' + \frac{1}{n}\ln.p \qquad 3.6$$

and a plot of ln.V against ln.p should produce a straight line whose slope is $1/_n$ and intercept, k', on the ordinate.

The Langmuir and Freundlich isotherms break down if the adsorbed layer is more than one monolayer thick. However, Brunauer, Emmett and Teller[58] expanded the Langmuir isotherm to include multi-layer adsorption,

$$\frac{V}{V_m} = \frac{k''x}{(1 - x)(1 + (k'' - 1)x)} \qquad 3.7$$

where,

$$x = \frac{p}{p^0} \qquad 3.8$$

and p^0 is the vapour pressure of the solute and k'' is a constant. The equation assumes that the heat of adsorption is constant over the whole surface coverage of the monolayer, secondary layers do not form until the monolayer is completed and that the heat of adsorption of secondary layers and above is equal to the heat of condensation of the adsorbate.

The potential theory of adsorption put forward by Polanyi[59] is based on a concept that cohesion forces attract molecules from distances somewhat greater than the molecular diameter. The intensity of attraction is known as the adsorption potential and is defined as the work done by the adsorptive forces in bringing a molecule from the gas phase to that given point. The adsorption potential rapidly decreases with distance and becomes zero within small finite spaces. A typical cross-section of the gas-solid boundary according to Polanyi's potential theory is shown in Figure 3.3.

The Dubinin concept of volume filling, reported by Hassler[60], offers some acceptable explanation for adsorption phenomena whereby the adsorption potential does not increase with adsorbent surface area. It suggests that the initial activation is such that the pores are sufficiently large to accept particular adsorbate molecules. On further activation, however, the pores become wider and the adsorbate molecules lose contact with certain surfaces of the pores and some are consequently desorbed. This loss offsets the gain from the increased surface area.

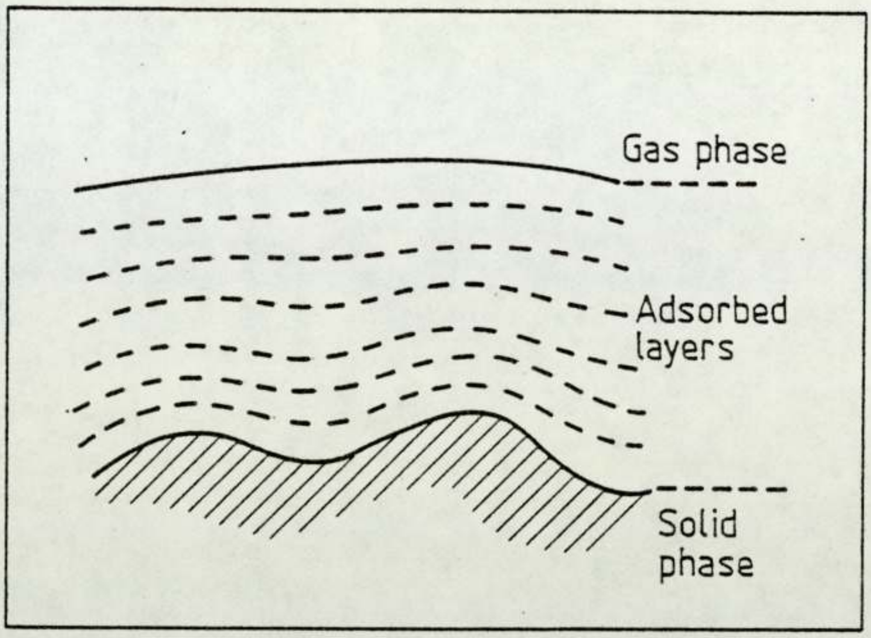


FIGURE 3.3 - THE STRUCTURE OF THE ADSORBED PHASE ACCORDING TO THE POLANYI POTENTIAL THEORY[55]

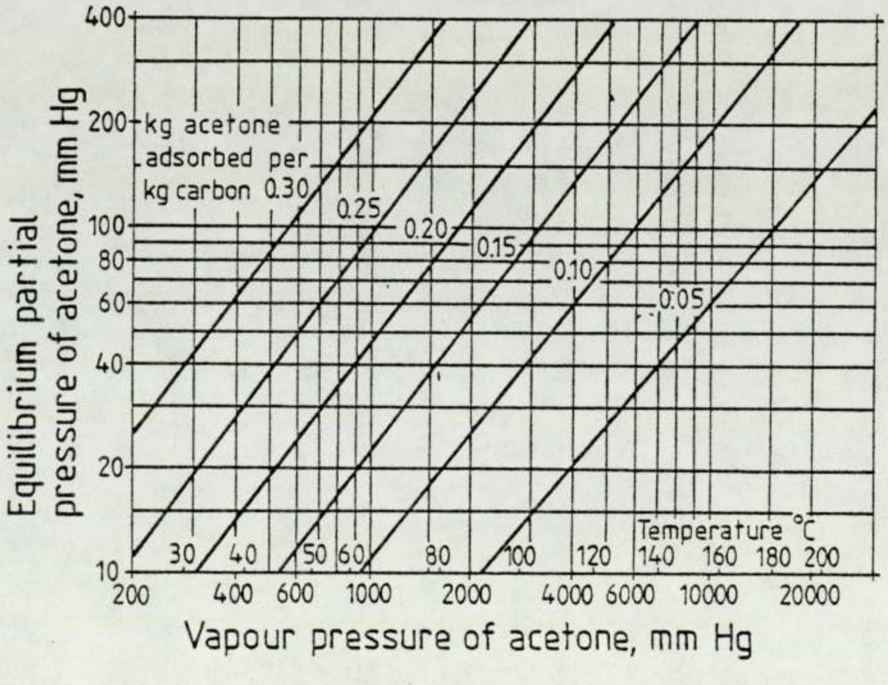


FIGURE 3.4 - REFERENCE SUBSTANCE PLOT OF EQUILIBRIUM ADSORPTION OF ACETONE ON ACTIVATED CARBON[69]

3.1.1.2 Multi-Component Adsorption

Although several theories and equations have been put forward for single component adsorption, multi-component equilibria normally exists in industrial applications. The adsorption equilibrium relationships of such systems are essentially extensions of pure component equations.

Markham and Benton[61] studied the adsorption of binary mixtures and interpreted their results on the basis of modifying Langmuir's equation which indicates that each gas in the mixture should be adsorbed to a smaller extent than if it were present alone in the same concentration. The Langmuir equation was further extended by Schay[62] which took into account molecular interaction in the adsorbed phase. Williams[63] proposed a correlation expressing the extent of adsorption in a n-component system to that of each of the pure components. More recently, Myers and Prausnitz[64] devised a technique for calculating the adsorption equilibria for components in a gaseous mixture using only pure component adsorption data.

3.1.2 Heat Effects

3.1.2.1 General

It is also necessary to study the effect of heat transfer in order to predict adsorber performance. In considering Equation 3.9 derived from first and second law thermodynamics[65] and the following relationship applies

$$\left(\Delta F = \Delta H - T\Delta S\right)_T \qquad 3.9$$

where, ΔF = change in free energy on adsorption

ΔH = enthalpy change on adsorption

ΔS = entropy change on adsorption

T = absolute temperature

The change in free energy is always negative in adsorption since the process proceeds spontaneously. There is also a decrease in entropy as the system is in a less orderly state prior to adsorption, consequently enthalpy change must be negative and so heat is liberated. The heat evolved is the heat of adsorption.

3.1.2.2 Heat of Adsorption

In order to desorb any material from an adsorbent, the heat of adsorption must be overcome. Therefore, the greater the heat of adsorption of the adsorbate-adsorbent system, the more heat is required to accomplish desorption.

Lamb and Coolidge[66] measured the quantity of heat evolved for the adsorption of a variety of vapours onto active charcoal. They stated that the heat of adsorption is made up of the heat of liquefaction of the particular vapour and a net heat of adsorption and both are of approximately the same magnitude. Later work by Keys and Marshall[67] showed that the rate at which heat is liberated during adsorption varies with time and generally the heat evolved during the initial stages of adsorption is greater than in subsequent periods.

Treybal[68] states that two heat terms are used in adsorption - integral and differential heat of adsorption. The isothermal integral heat of adsorption is defined as the total heat liberated in adsorbing from zero loading to some final loading at constant temperature. The differential heat of adsorption represents the change in integral heat of adsorption with change in loading.

Othmer and Sawyer[69] have indicated that the heat of adsorption can be estimated from plots similar to the type shown in Figure 3.4. The slope of an isotere may be given by

$$\frac{d \ln p}{d \ln P^0} = \frac{(-\overline{H})\ M}{\lambda_r\ M_r} \qquad 3.10$$

where $\overline{H}$ represents the differential heat of adsorption, M and M_r the molecular weights of the vapour and reference substance respectively, and λ_r the latent heat of vapourization of the reference substance.

If the differential heat of adsorption is computed for each isotere at constant temperature, the integral heat of adsorption at this temperature can be determined from the relation

$$\Delta H_a' = \int_0^W \overline{H}\ dW \qquad 3.11$$

where, $\Delta H_a'$ represents the integral heat of adsorption referred to vapour adsorbate per unit mass of adsorbent and, W the concentration of adsorbate per mass of adsorbent. If $\overline{H}$ is plotted against W, the integral can be evaluated graphically by calculating the area under the curve. The integral heat of adsorption referred to liquid adsorbate per unit mass of adsorbent, ΔH_a, is

$$\Delta H_a = \Delta H_a' + \lambda W \qquad 3.12$$

3.2 Mass Transfer

3.2.1 General Considerations

In addition to equilibrium data for the adsorbate-adsorbent system, adsorption rate should also be considered. As in adsorption equilibria, there is no one single approach which is satisfactory and completely versatile in expressing adsorption kinetics. However, two methods, termed 'micro' and 'macro' approaches are available.

Mass transfer coefficients and suitable diffusion equations are used in the micro approach and each of the resistances to mass transfer must be considered. McKetta[53] states that although mass transfer coefficients give some insight into the adsorption mechanism, they are difficult to evaluate.

In the macro approach, a total resistance to mass transfer is expressed in terms of the amount of adsorbent which has not been used. This method, using a mass transfer zone concept was originally put forward by Michaels[70] and is known to be simple and effective in studying adsorption kinetics in fixed bed systems.

For completeness, however, both methods are described below.

3.2.1.1 Micro Approach

The micro approach to the kinetics of adsorption involves the prediction of the step which most inhibits the transport of an adsorbate molecule to the active sites of the adsorbent, this being the rate-limiting step. It describes the step which controls the rate at which adsorption proceeds. Adsorption may therefore be discussed in terms of rate equations.

The process of adsorption takes place in a number of stages and each one could represent the limiting stage. Weber[71] suggested a three stage system. In the case for the adsorption of an organic solvent in the vapour phase onto activated granular charcoal, the organic molecules are transferred from the bulk carrier gas stream to the external surface of the charcoal. The flux of the adsorbate across the external gas film surrounding the adsorbent can be expressed in terms of a mass transfer coefficient as shown by the relation

$$k_c(y - yp) = -D_e \frac{\partial yp}{\partial r} \qquad 3.13$$

where y and yp represent the gas phase and pore gas concentrations respectively, D_e is the effective diffusivity, r is the radial parameter and k_c is the mass transfer coefficient. The latter can usually be computed from correlations such as that of de Acetis and Thodos[72], for fixed beds.

The following step is intraparticle diffusion whereby organic solvent molecules are transferred from the external surface of the charcoal to its internal surface by some diffusive mechanism. The rate of diffusion within a porous material can be expressed by means of Fick's Law and assumes a spherical adsorbent particle -

$$\frac{\partial y}{\partial t} = \frac{1}{r^2}\frac{\partial}{\partial r}\left(D_e\, r^2 \frac{\partial yp}{\partial r}\right) \qquad 3.14$$

The type of diffusion taking place within the particles' pores depends on the relative size of the pore diameter and the mean free path of the vapour under the conditions which exist in the pores. At low pressures, or in small diameter pores, that is, pores with diameters appreciably less than 10^{-7}m[54], Knusden[73] showed the diffusion coefficient to be given by

$$D_k = 1.33r_p\left(\frac{2RT}{\pi M}\right)^{0\cdot5} = 0.66r_p\overline{U} \qquad 3.15$$

where D_k represents the Knusden diffusion coefficient, r_p the pore radius and $\overline{U}$ the mean molecular velocity. In larger pores, or at high pressures, most collisions are between molecules and the diffusive mechanism is the same as in the bulk gas. Values of such diffusion coefficients can be obtained from the literature[74,75].

Finally, in physical adsorption, such as is the adsorption of organic molecules onto active carbons, the actual adsorption rate is extremely rapid and is regarded as not to influence the overall kinetics.

The relative effects of interparticle and intraparticle mass transfer can be evaluated from the Nusselt Number[54]. The ratio of the resistance to mass transfer to that in the external gas film is defined by the Nusselt Number and generally shows that most of the resistance is in the solid. Vermeulen[76] has shown that under isothermal conditions the diffusional resistances may be combined to give one overall resistance to mass transfer.

3.2.1.2 Macro Approach

The concept of the macro approach is built around the mass transfer zone and its behaviour during adsorption. The adsorption wave and its movement during adsorption with respect to the uptake of an organic solvent vapour from air onto active carbon is first described, followed by Michaels'[70] approximation for total mass transfer resistance.

3.2.1.2.1 The Adsorption Wave

When an air/solvent mixture is passed through a bed of fresh activated charcoal, initially, most of the adsorbate is taken up at the inlet part of the adsorbent bed and the fluid moves towards the column outlet with little further adsorption taking place. Adsorption takes place further along the charcoal bed once the inlet part of the adsorbent becomes saturated. The bulk of adsorption therefore takes place over a relatively narrow adsorption zone. This is the mass transfer zone.

As the air carrying the solvent continues to flow, the adsorption zone moves up the column as a wave. The longer the adsorption wave, the greater the resistance to mass transfer and Lukchris[77] states that generally such waves are 'S' shaped. In contrast, as shown in Figure 3.5, the ideal adsorption wave would be vertical dividing the equilibrium zone and unused bed, the concentration changing instantaneously from zero to the

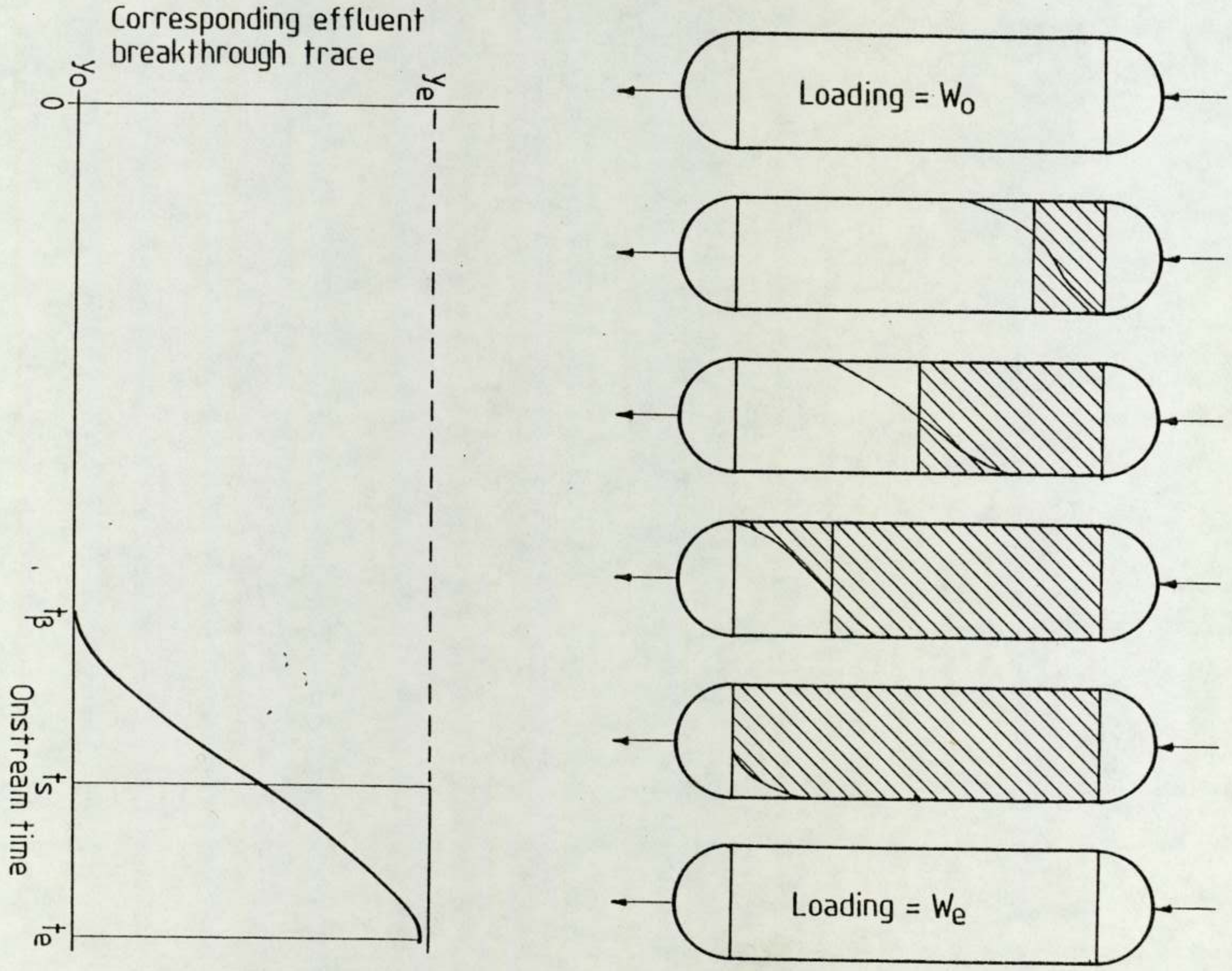


FIGURE 3.5 - THE ADSORPTION WAVE (STABLE MASS TRANSFER FRONT RELATIVE TO STOCHIOMETRIC FRONT)[53]

inlet concentration. The length of the adsorption zone would therefore be zero.

With reference to Figure 3.5, when approximately half of the bed is saturated, the effluent concentration essentially remains zero and does not rise to an appreciable value, y_B, until the adsorption zone has just reached the column outlet. The system has now attained its break-point. The outlet concentration continues to rise to y_S beyond which little adsorption takes place as the charcoal bed is considered to be practically at equilibrium with the inlet solution.

The portion of the effluent concentration curve between positions y_B and y_S is termed the break-through curve. Its shape is influenced by a number of factors including the fluid velocity, adsorber length and the concentration of the solute in the feed[78]. In an ideal situation, as shown in Figure 3.5, the adsorption would be infinitely rapid and the break-through curve would be a straight, vertical line.

3.2.1.2.2 Rate of Adsorption in Fixed Beds

Several assumptions must be made before the macro approach suggested by Michaels[70] can be adopted. Firstly, it assumes isothermal adsorption from dilute feed mixtures and it is limited to cases where the equilibrium adsorption isotherm is concave to the solution concentration axis. In addition to this, the height of the adsorption zone is assumed to be constant as it moves through the adsorber column and the height of the adsorbent bed is large relative to the height of the adsorption zone.

The total resistance to mass transfer is expressed in terms of the amount of adsorbent bed which remains unused at break-point. The degree of saturation of the adsorbent, ι, is given by the relation

$$\iota = \frac{D - fD_a}{D} \qquad 3.16$$

where D and D_a represent the height or depth of the adsorbent bed and adsorption zone respectively, and f, fractional ability of the adsorption zone to adsorb solute.

This analysis, however, is also restricted to the constancy of the overall gas phase mass transfer coefficient for concentrations within the adsorption zone. Collins[79] derived an expression for the length of unused bed, L, shown by Equation 3.17.

$$L = D - \left(\frac{G_S \, t_B \, (Y_O - Y_O^*)}{\rho_s \, (W_e - W_o)} \right) \qquad 3.17$$

where G_S is the mass velocity of unadsorbed gas based on the cross-section of the column, t_B, the time to break-through, ρ_s, the apparent density of the adsorbent bed, Y_o, the initial concentration of the solute in the fluid in terms of mass solute/mass solvent, Y_o^*, the fluid concentration in equilibrium with W_o, the initial adsorbent loading.

3.3 Factors Affecting Adsorption

It has already been pointed out that the performance of an adsorptive system can be evaluated using mass transfer zone characteristics. The adsorption dynamics in addition to the length and shape of the mass transfer zone are influenced by a number of factors. The individual and combined effects of such factors is now described with respect to vapour phase adsorption onto activated charcoals.

The size and shape of charcoal particles influence both the pressure drop across the adsorbent bed and the rate of diffusion of the species to be adsorbed. A low pressure drop will exist if relatively large, uniform, spherical particles are used, but since mass transfer decreases with increasing particle size, adsorption onto smaller particles is more efficient, and is in fact preferred even at the expense of a higher pressure drop[80].

The effectiveness of activated carbon as an adsorbent is enhanced by its large surface area. Brunauer, Emmett and Teller[58] developed a method of measuring internal surface area by studying the adsorption of nitrogen onto activated carbon at low temperatures. Heat of wetting, that is, the amount of heat liberated when a solid is submerged in a liquid, gives some indication of the solid's adsorbability and generally, the higher the heat of wetting the greater the adsorptive potential of the solid[81].

However, although the surface area of an active carbon may be large, the pores inherent in the adsorbent material may only be small enough to accommodate small molecules. Therefore, the adsorptive property of the porous substance may be small with regard to large molecules. Adsorbent pore sizes have been classified by Dubinin[82] who stated that there were three different types - macropores, transitional pores and micropores. These are shown diagrammatically in Figure 3.6. The effective radii of each class of pore is summarized in Table 3.1 and it is shown in the Table that most of the surface area of the charcoal adsorbent lies in its micropores.

Type of Pore	Effective Radius - Å	Volume m^3/kg	Surface Area m^2/kg
Macropore	5,000-20,000	0.0002 -0.0008	500- 2,000
Transitional Pore	30- 2,000	0.00002-0.0001	20,000-70,000
Micropore	< 18- 20	0.00015-0.0005	95% total specific area

Table 3.1 - Pore properties of activated carbons

The techniques available for determining pore structure are generally helium density, mercury density, nitrogen adsorption and mercury penetration and the method selected depends on the pore size range[83]. For

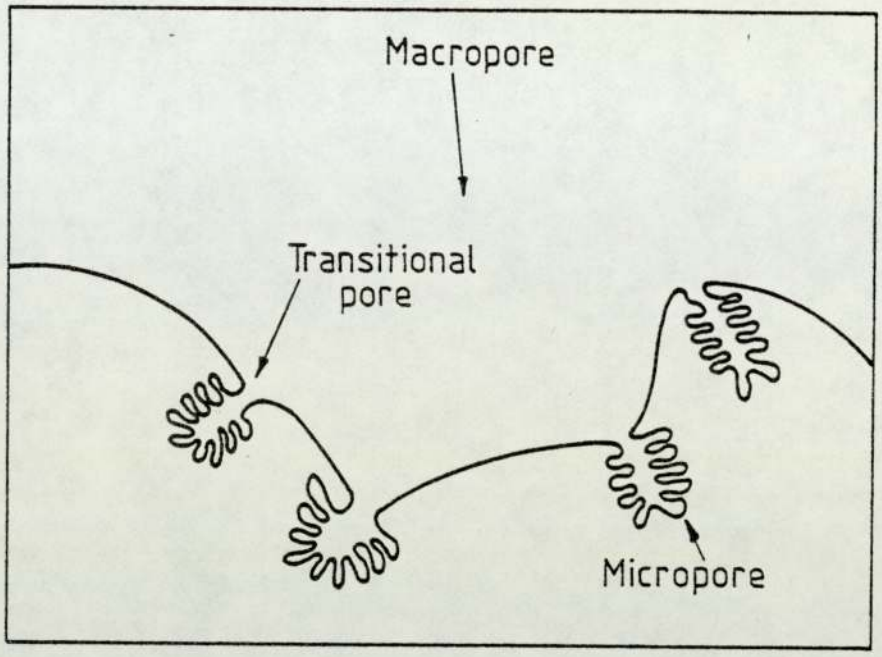


FIGURE 3.6 - THREE PORE MODEL[82]

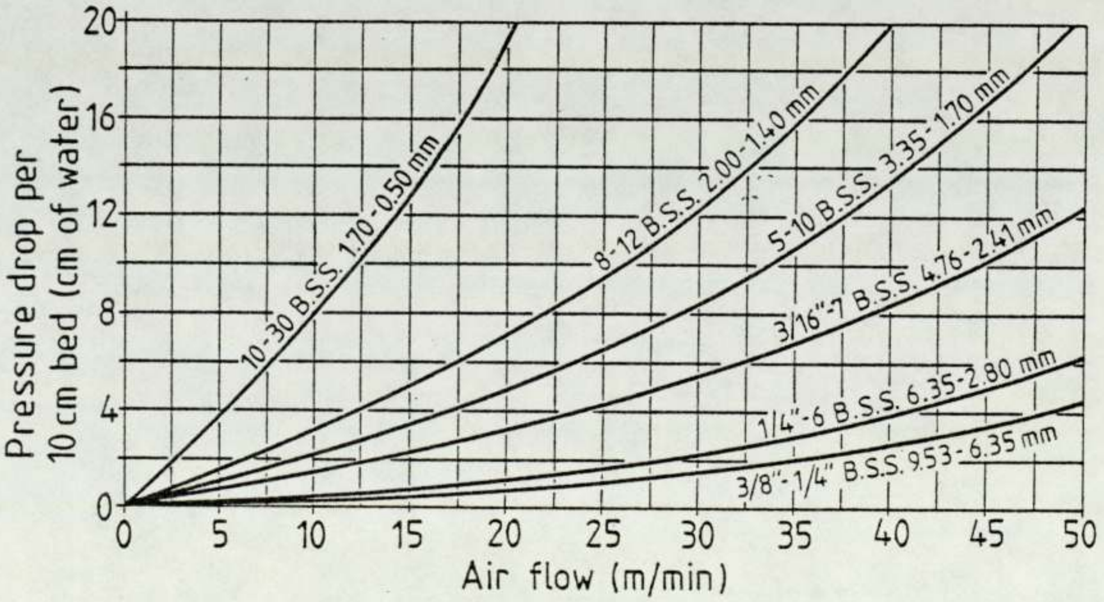


FIGURE 3.7 - RESISTANCE TO AIR FLOW OF BED OF GRANULAR ACTIVATED CARBON[22]

example, the mercury porosimetry method is useful in obtaining information on pore size distribution in the range of radii 15 - 150 Å[84].

The effectiveness of an adsorbent system is not only dependent on the surface area or pore size range of the adsorbent, but the orientation of the adsorbed molecule and the shape of the pore. An aliphatic acid molecule lying flat on its surface covers more carbon atoms than if attached at one end[85]. Bailey[86] suggested a greater uptake of benzene than carbon tetrachloride by active charcoal adsorbent to some degree resulted from the pore shape inhibiting entry for the organic chloride molecules.

The length of the mass transfer zone is directly proportional with velocity, thus the unsaturated zone is lengthened at high velocities. The pressure drop is also greater at higher gas velocities and Figure 3.7 shows the relationship between pressure drop and gas velocity for different charcoal particle sizes[22]. For example, at a superficial gas velocity of 0.134 m/s, the pressure drop per 0.10m bed is found to be 0.002m and 0.063m of water for particle size ranges of 0.00953m to 0.00635m and 0.00170m to 0.0005m respectively. In contrast to this, if discs of cloth are placed in a column such that its bulk density is 300 kg/m^3, the pressure drop per 0.10m of column would be as high as 0.44m of water if the vapour flows through the cloth. (Calculations supporting this are included in Appendix 1.) Lower pressure drops in the order of 0.005m of water are attained if the gas stream carrying the adsorbate flows parallel to charcoal cloth supported on a wooden former, but in adopting this arrangement the equivalent bulk density of the charcoal cloth is greatly reduced.

It is important that the length or height of the adsorber should be at least the length of the mass transfer zone of the key component being adsorbed. Moreover, by increasing the minimum bed depth, more than a proportional increase in capacity is achieved. The depth of the adsorbent bed is normally designed to the maximum pressure drop allowed[80].

Figure 3.1 shows that adsorption decreases with increasing temperature. The effect of increased pressure has little effect on the adsorption of the more adsorbable vapours, but generally, the uptake of less adsorbable molecules is slightly increased at higher pressures. Changes in pressure also affect the relative amounts of material adsorbed from mixtures[87].

As has been discussed previously, the adsorptive capacity of adsorbents is directly proportional to the concentration of the adsorbate, which, in turn is inversely proportional to the length of the mass transfer zone. Thus, everything else being equal, in the case for the adsorption of organic solvent molecules onto an activated carbon bed, a deeper bed would be required for a lower concentration of the organic solvent with equal efficiency than to remove the same material at higher concentrations.

Some portion of all gases present will be taken up by the adsorbent surface and since the gases are competing for adsorption sites, the adsorptive capacity for the particular adsorbate is reduced. Under ambient conditions only a small quantity of air ($1.0 - 2.0 \times 10^{-4}$ m^3/kg) is adsorbed onto most commercial adsorbents[80], and is therefore generally regarded as negligible. Moisture and carbon dioxide, however, have a more significant effect. For example, Table 2.5 shows that the presence of moisture decreased the adsorptive capacity of both nutshell charcoal and charcoal cloth, but the reduction in solvent uptake by the granular charcoal is much greater[33,34,35,36,37].

If a mixture of two or more gases is to be removed from a carrier vapour stream, the more strongly adsorbed contaminant in the pure state preferentially adheres to the adsorbent surface. However, the amount of material adsorbed per unit weight of the porous solid is less than if the component in question were in the pure state at the same partial pressure[80].

It is known that some activate carbons decompose, react or polymerize when brought into contact with some adsorbents. For example, nitric oxide in an air stream is converted to nitrogen dioxide in the presence of activated charcoal[80]. The adsorptive capacity of active carbons is diminished if there is polymerization on its surface and regeneration may not be able to be carried out by conventional procedures. Additionally, regeneration using steam may also cause some decomposition of the adsorbate[50].

It is stated in Section 2.4.2.2 that the number, type and arrangement of adsorbers affects the overall efficiency of a solvent recovery plant and a further factor which influences the capacity of an adsorptive system is effected by the operating procedures. For example, it is normally desirable to have the steam for regeneration flowing in a direction opposite to that of the fluid[80]. In doing so, the effluent end of the bed is kept free from impurity because if desorption is carried out co-currently to adsorption, some of the material initially desorbed from the equilibrium section will be re-adsorbed on the unused zone where it has to be desorbed a second time. Adsorbers are normally operated periodically and the inlet concentration of the contaminant(s) in the air varies under such conditions. This results in the capacity of the adsorptive system being reduced.

3.4 Regeneration

During adsorption the free energy of the system is decreased and so energy must be provided to drive off the adsorbate from the adsorbent. The optimum conditions for regeneration must be applied in order to give the highest adsorbent efficiency and life with the minimum energy input during desorption. There are five basic techniques for regenerating a loaded bed of adsorbent[53,54], and these are discussed in Section 3.4.1. The kinetics of regeneration using low pressure steam are briefly described in Section 3.4.2.

3.4.1 Regeneration Techniques

3.4.1.1 Thermal Swing Desorption

Thermal swing desorption, the most commonly used regeneration method, involves driving off the adsorbate by raising the adsorbent bed temperature. The temperature can be increased by circulating a hot desorbable vapour through the bed, heating with internal coils or a combination of both. This method of regeneration decreases the equilibrium adsorbate loading for a given adsorbate vapour pressure.

3.4.1.2 Pressure Swing Desorption

Pressure swing desorption can be accomplished at constant temperature by carrying out desorption under pressure and regeneration at a lower total system pressure. The pressure swing cycle is relatively simple, it can be changed rapidly, heating and cooling steps are eliminated and a pure product is recovered since no separation of the desorbate and stripping agent is necessary.

3.4.1.3 Purge Gas Stripping

Purge gas stripping is achieved by passing an essentially non-adsorbed gas through the adsorbent bed. The purge gas removes the desorbate from the adsorbent surfaces, thus keeping the partial pressure of the component over the bed below its equilibrium value. The amount of material desorbed is influenced by the temperature and total pressure of the operation.

3.4.1.4 Displacement Desorption

A displacement desorption cycle is one whereby a more strongly adsorbed adsorbate is used to displace the original adsorbate. This regeneration technique is often used to desorb a temperature sensitive material, but a further desorption step is needed to desorb the displacement substance before commencing adsorption on the next cycle.

3.4.1.5 Combination Desorption

The removal of solvents from activated charcoals using low pressure steam is an example of a combination regenerative cycle. Thermal swing, purge gas and displacement desorption techniques are all involved in the regeneration process.

3.4.2 Regeneration Kinetics

Since the research project is involved with the adsorption and desorption of solvent vapours with respect to activated charcoals, the dynamics of regeneration using low pressure steam only is described.

Rosen[88] states that desorption is identically opposite that of adsorption. The amount of steam required for the desorption of a particular material directly depends on the heat of adsorption of the component onto the porous substance, and on the heat given up to the surroundings. The regeneration rate is enhanced by insulation which minimizes the heat lost to the adsorber walls and other auxiliary equipment.

Desorption kinetics are also influenced by the rate heat is transferred across the external gas film from the bulk gas stream to the external surface of the adsorbent and by solid and gaseous conduction from the external surface to the internal surfaces of the porous material[54].

The amount of steam needed initially to remove unit mass of solvent adsorbed is lower than that required in subsequent periods. Hassler[89] states that several theories have been put forward regarding changes in the heat of adsorption. Generally, more heat is released during the initial stages of adsorption. Evidence suggests that in multi-layer adsorption, this phenomena is a result of stronger forces existing in the monolayer than in subsequent layers. Other ideas propose that molecules are initially adsorbed in crevices and capillaries where bonds are formed on several sides and at the adsorbents' more active sites.

Since steam requirements are the major running costs in operating a solvent recovery plant, any steam savings would be very beneficial. It is therefore intended to investigate for any reduction in steam requirements and desorption rate in studying the removal of organic solvents from activated charcoal cloth over granular charcoal.

3.5 De-Activation

De-activation can be defined as the deterioration in the initial performance level of an adsorbent[53]. This phenomena is often caused by the accumulation of contaminants on the adsorbent surface which is not removed during normal regeneration. The resistance to mass transfer can also be greatly increased if the contaminants build up in the larger pores of the adsorbent. De-activation can also result from chemical changes occurring with trace contaminants which reduce surface activity. It is therefore important to blow out dirt from auxiliary pipe-lines before start up so that it is not flushed onto the adsorbent bed.

It is difficult to estimate the number of adsorptive-desorptive cycles an adsorbent can undergo before it needs replacing. However, some information can be gained from laboratory experiments whereby a sample of adsorbent is subjected to thousands of process cycles in a simulated process stream or by monitoring the actual performance of the adsorbent over long time periods in actual service. The life factors of an adsorbent greatly depends on the conditions of the adsorptive system.

CHAPTER 4

BATCH RIG OPERATIONS AND RESULTS

PLATE 4.1

GENERAL VIEW OF EXPERIMENTAL APPARATUS

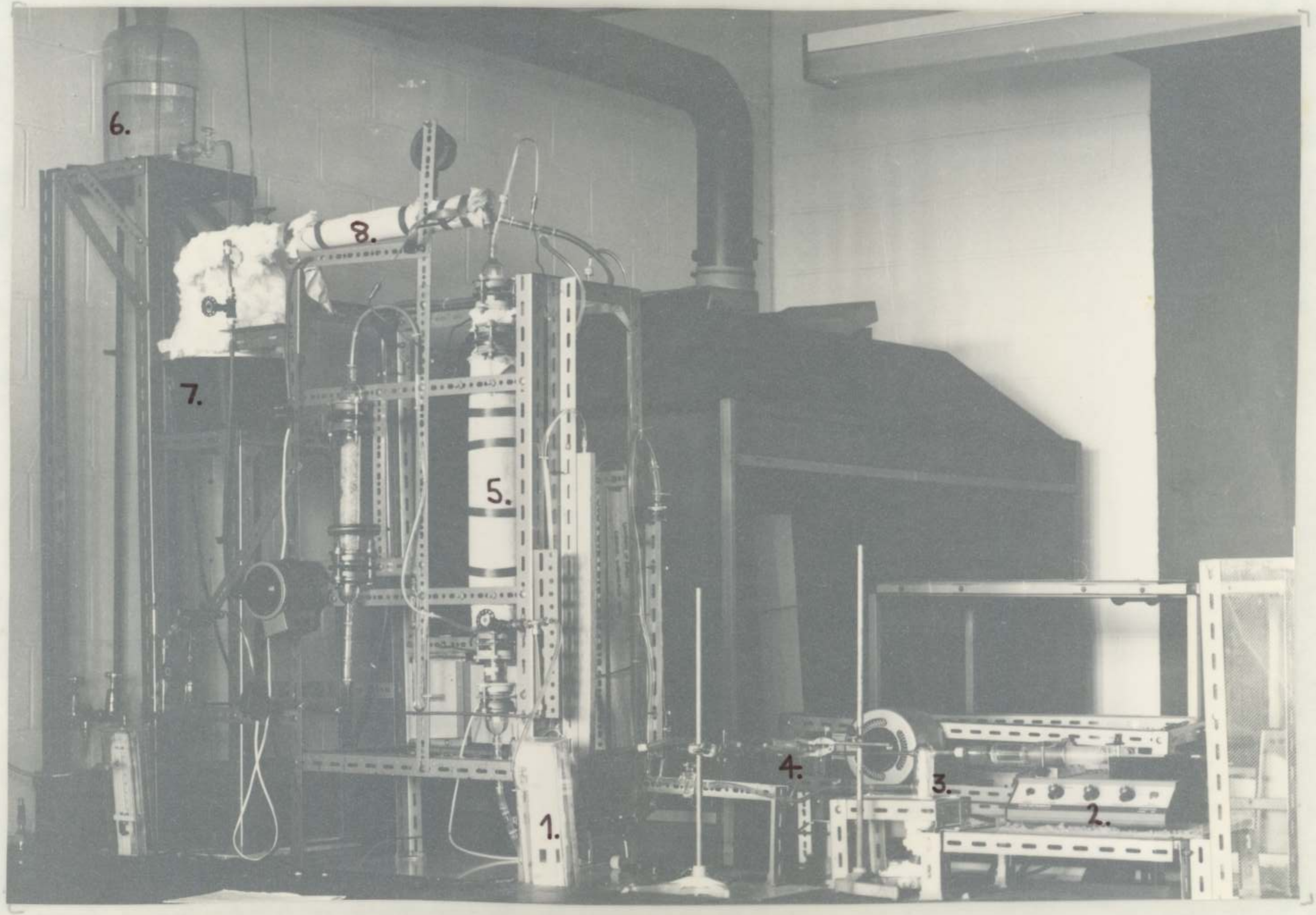
USED IN THE BATCH OPERATIONS

1	Hygrometer
2	Syringe Pump
3	Hot 'Glass Finger'
4	Solvent-Air Mixing Unit
5	Adsorber
6	Water Reservoir
7	Boiler
8	Superheater

PLATE 4.2

BATCH RIG SET-UP REGENERATION

1 Water Reservoir
2 Boiler
3 Superheater
4 Adsorber
5 Outlet Tube Directing Stripped Vapour to Condenser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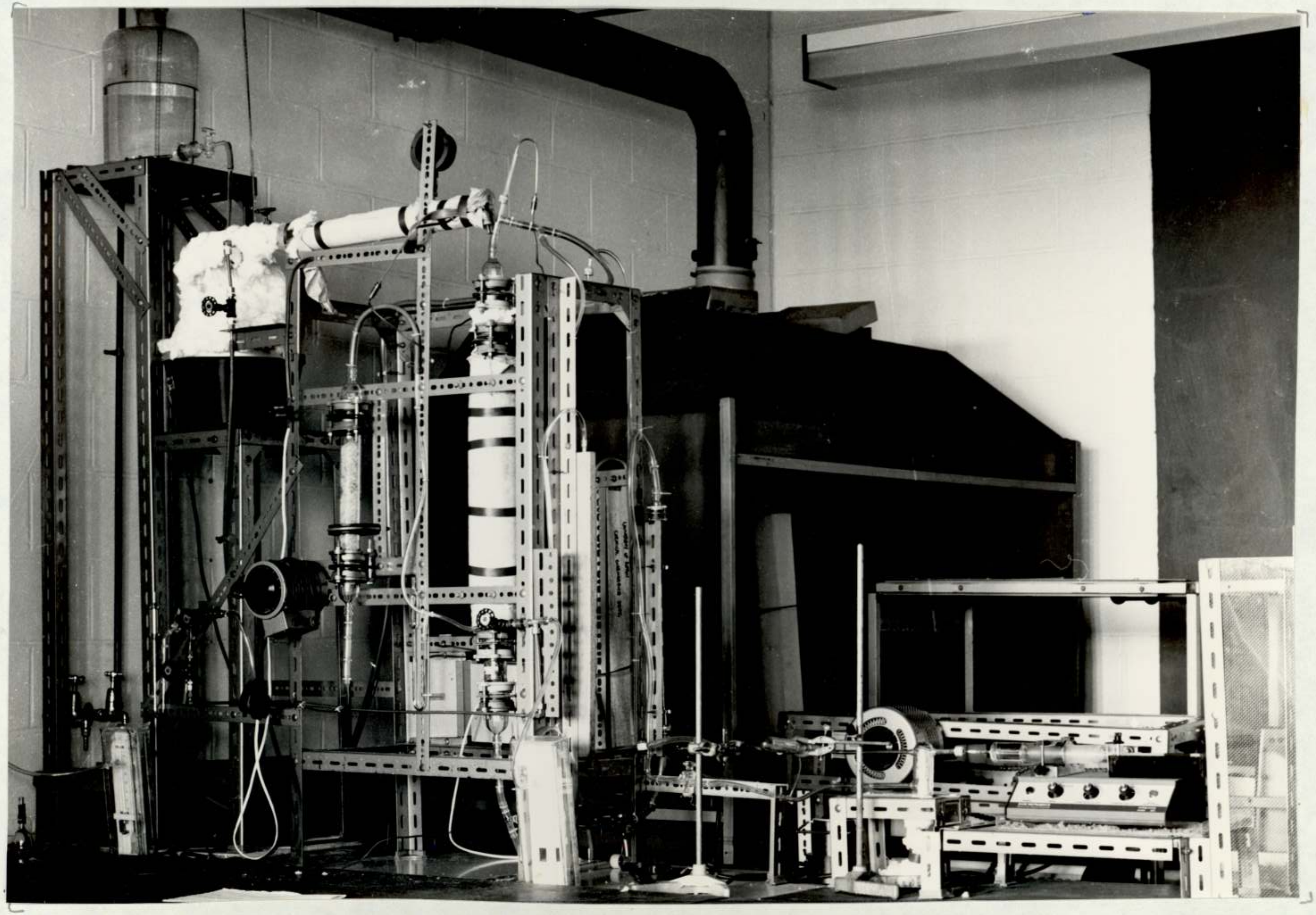
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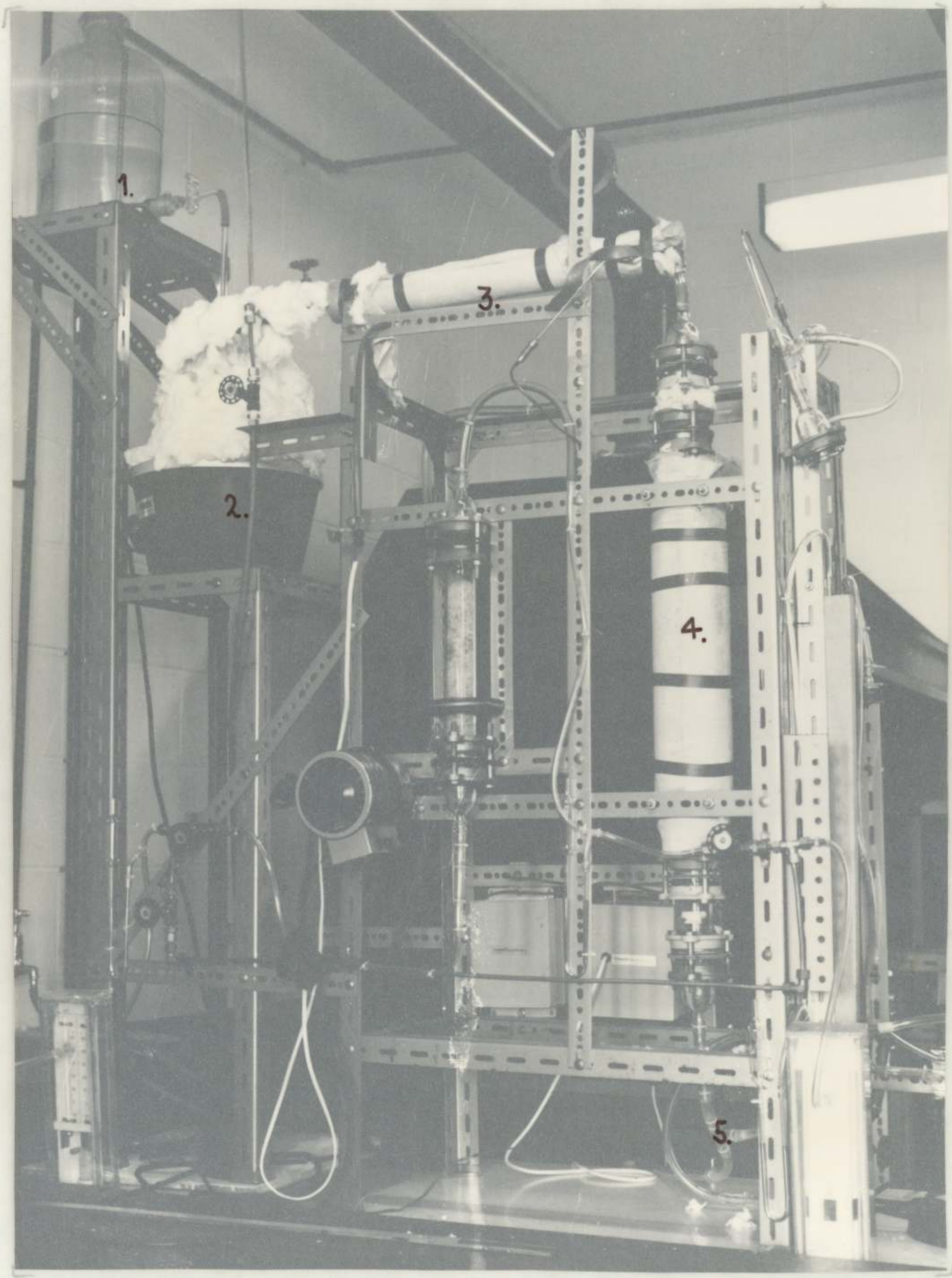
Chemical Engineering Dept.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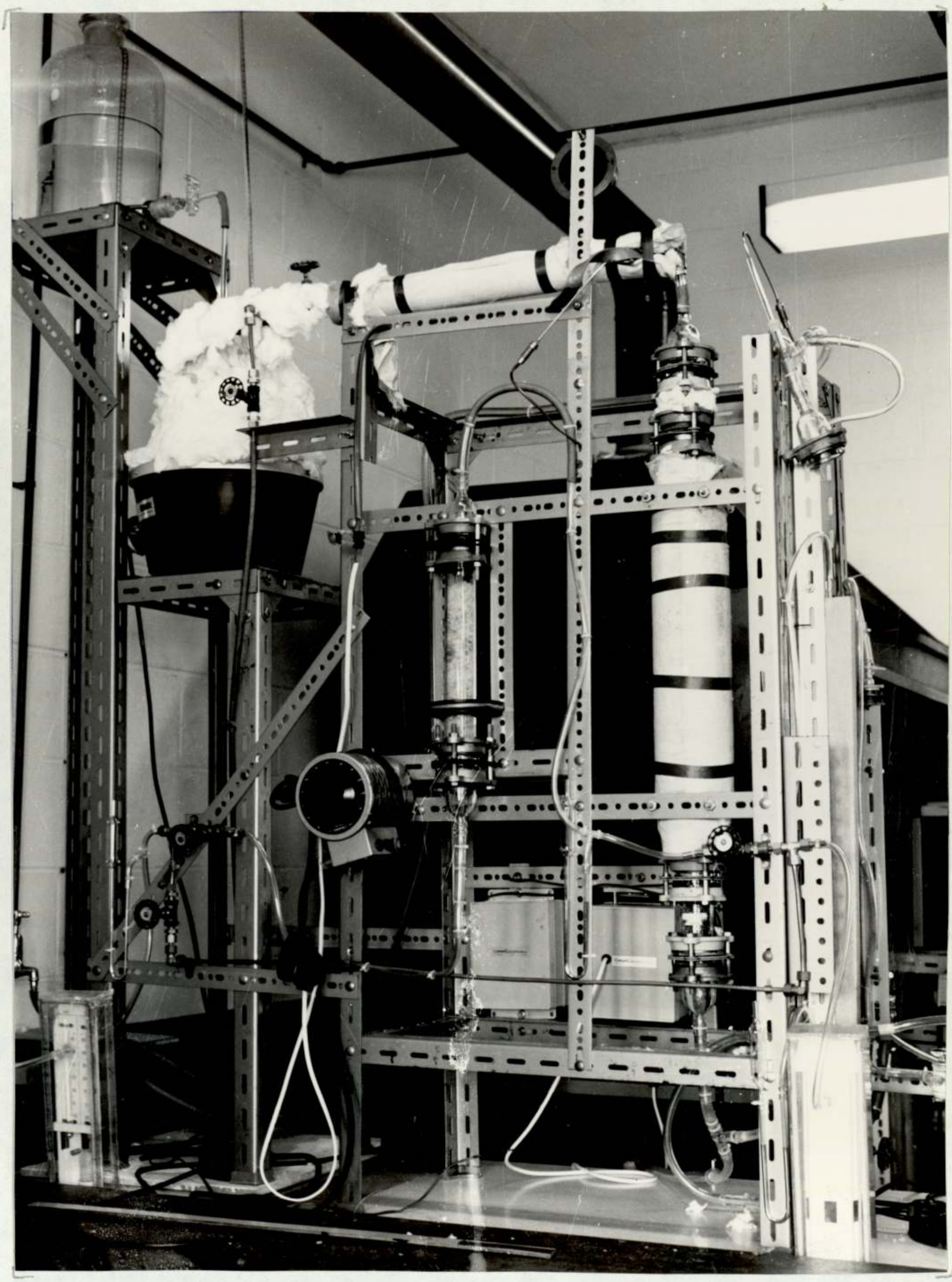


4 BATCH RIG OPERATIONS AND RESULTS

This Chapter describes the investigations into the batchwise recovery of perchloroethylene and toluene vapour from an air stream using activated charcoal cloth.

The experimental objectives are outlined in Section 4.1 and are followed by a description of the experimental facilities used. Details of the experimental conditions for both single and cyclic adsorption-desorption processes are included in Section 4.3, succeeded by a discussion of the results obtained.

4.1 Experimental Objectives

Firstly, the extent of adsorption of perchloroethylene and toluene vapour onto virgin activated charcoal cloth is studied. The adsorption is continued until the charcoal cloth is saturated, that is, when the system has attained equilibrium. Results from these experiments are compared with the equilibrium adsorptive potential of charcoal cloth which has undergone a number of regenerations.

An equilibrium adsorption curve is a plot of the mass ratio of solute to solvent against a mass ratio of adsorbate to adsorbent. It provides data from which a model predicting the parameters for the system can be developed for scale up purposes. Therefore, relationships are drawn between the organic solvent concentration in the air and that adsorbed on the charcoal cloth surface at equilibrium.

Breakthrough curves, which are plots of the effluent concentration against time, and equilibrium curves are used in determining a total resistance to mass transfer expressed in terms of the amount of adsorbent bed unused at breakpoint. Additionally, breakthrough curves provide information necessary for evaluating the length of unused adsorbent bed at breakpoint and an overall bed height. These adsorption waves were drawn for each experimental run described in Section 4.3.

The superficial velocity of the fluid through the column affects adsorption and the consequence of varying the velocity for a constant solvent concentration is examined.

Once the cloth becomes saturated with solvent it is regenerated using steam. The effectiveness and efficiency of the solvent recovery is assessed by regularly monitoring the amount of steam and solvent vapour condensed for each experiment. Finally, comparisons are made between the steam requirements and quantities of solvent recovered in single and cyclic adsorption-desorption operations.

4.2. Batch Rig Description

The batch rig is firstly outlined in general terms followed by a more detailed account of the equipment used. The analytical techniques employed during adsorption and regeneration are described in Section 4.2.2 and, finally, the design of the batch system with regard to safety is presented.

4.2.1 General Description

The experimental equipment used for the batch operations is indicated in Plate 4.1 and is shown in a schematic form in Figure 4.1.

Liquid solvent is vapourized over a glass 'hot finger' as depicted in Figure 4.2. The organic vapour is carried by an air stream to the adsorption column via a mixing device. The solvent laden air stream flows up the adsorber, parallel to the charcoal cloth which is wound on

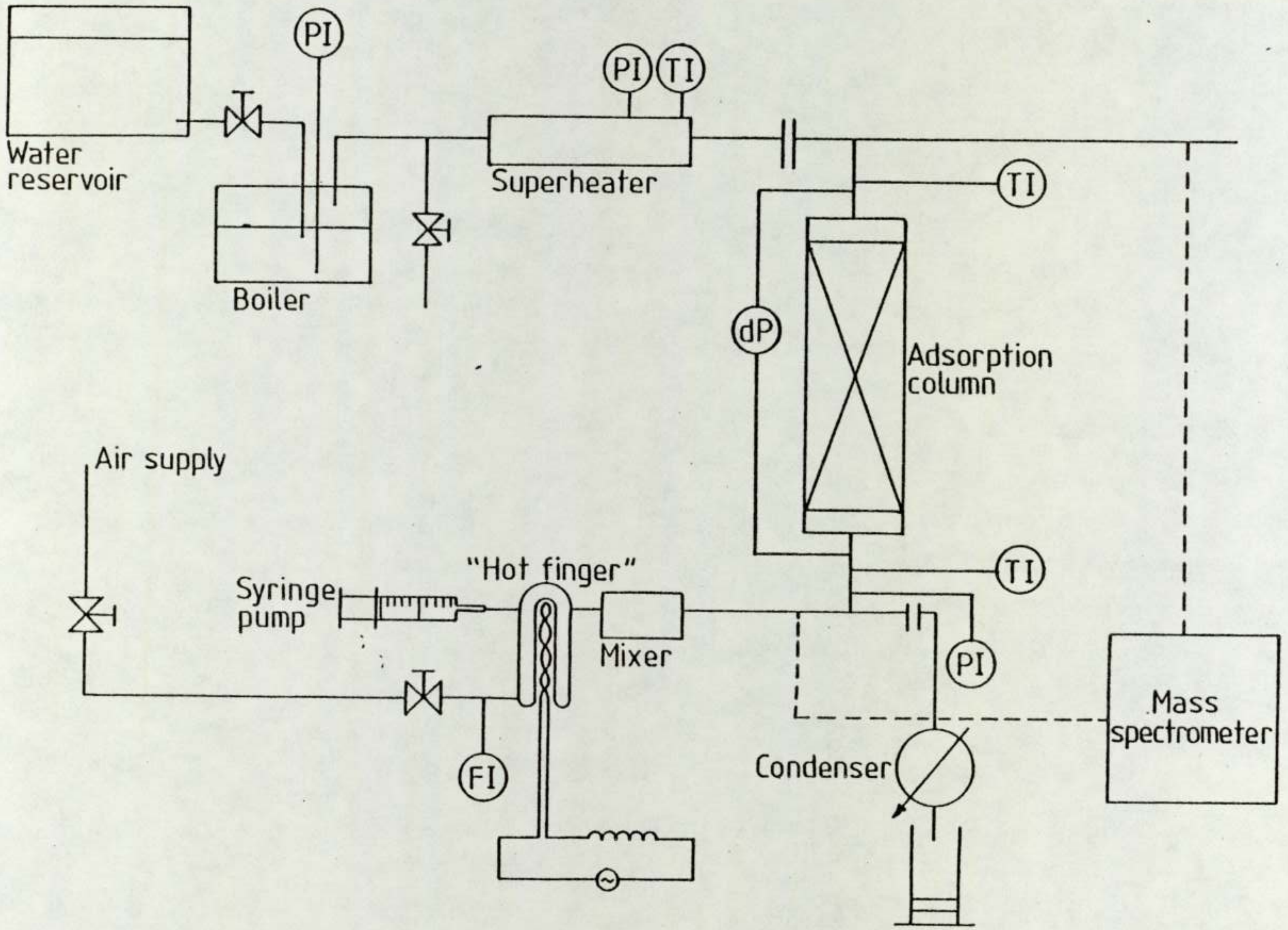


FIGURE 4.1 - SCHEMATIC DIAGRAM OF EXPERIMENTAL APPARATUS USED FOR THE BATCH OPERATIONS

a wooden former and supported by a nylon mesh. The activated charcoal cloth takes up the organic solvent vapour and the exhaust from the column is extracted from the laboratory via a fume hood. The adsorption process is continued until the cloth is saturated.

Once equilibrium has been reached, the charcoal cloth is regenerated by low pressure steam. The set-up of the batch rig for regeneration is shown in Plate 4.2. Steam, available in situ, descends the adsorber where it strips the organic solvent from the cloth. Finally, the steam-solvent vapour mixture is condensed before being collected in a flask.

4.2.1.1 Solvent-Air Mixing Section

Air, to carry the organic vapour, is available from a pipeline in the laboratory. Its volumetric flow is measured by a metric 7A rotameter and can be varied between rates of 1.66×10^{-5} $m^3.s^{-1}$ and 1.83×10^{-4} $m^3.s^{-1}$ by a plug valve immediately preceeding the flow indicator. The relative humidity of the air is determined from a wet and dry bulb hygrometer enclosed in a perspex box through which the air flows.

A syringe pump, Model 351 ex Sage Instruments, which can deliver liquids at rates between 2.5×10^{-7} $m^3.s^{-1}$ and 1.0×10^{-4} $m^3.s^{-1}$ is used to infuse the liquid solvent onto the glass 'hot finger'. The 'hot finger', used to vapourize the solvent, is heated by 1.95m of heating wire which has a resistance of 150 $\Omega.m^{-1}$. The heating wire is pushed up inside the 'glass finger', and the ends of the wire are connected to the terminals of a 2A Torovolt with an output voltage ranging from 0 - 270v.

The set-up shown in Figure 4.3 is used to mix the air and solvent vapour before it is passed through PVC tubing to the base of the adsorber.

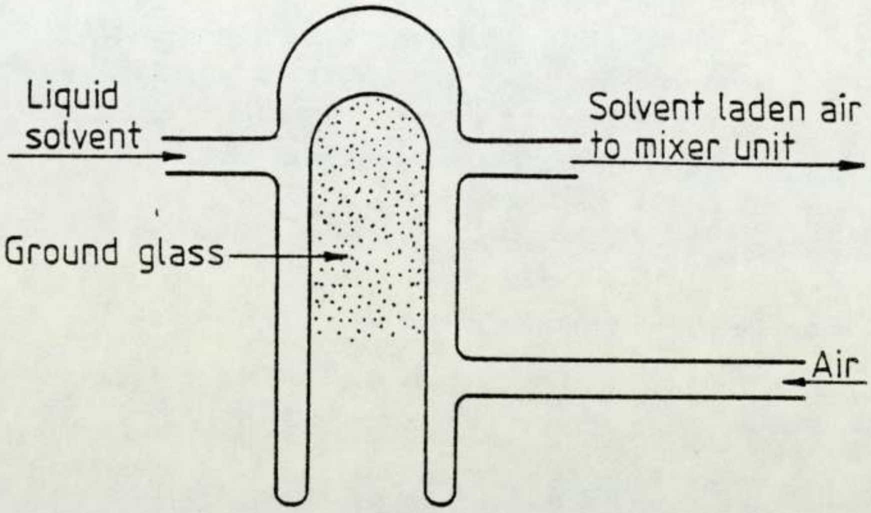


FIGURE 4.2 - THE HOT GLASS FINGER SYSTEM

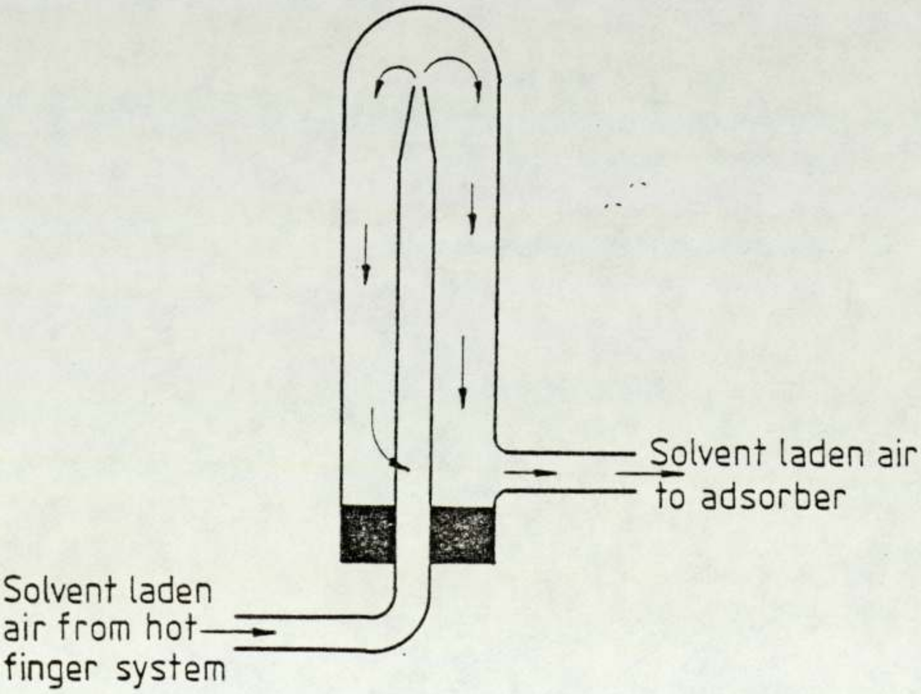


FIGURE 4.3 - MIXER UNIT

4.2.1.2 The Adsorber

The adsorber itself is a glass cylinder 0.457m in height and 0.38 x 10^{-1}m in diameter. It is insulated by fibre glass so that adsorption and regeneration are carried out with minimal heat losses. Filters, consisting of two pieces of nylon mesh between which is a cylinder of knitmesh have been fitted at each end of the column. The use of these filters is primarily to prevent contamination by fine solid particles.

The cloth, generally measuring 0.72m by 0.60m and weighing 0.45 x 10^{-1}kg, is wound spirally on a 0.524 x 10^{-2}m diameter wooden former and supported by a 22g nylon mesh. The mesh also promotes mixing of fluid across the cloth.

The absolute pressure and the pressure drop across the adsorber are obtained from water manometers and thermocouples are used to measure the temperature of the gas at the column inlet and outlet.

Finally, the outlet vapour from the adsorber pass through PVC tubing to a fume hood where it is discharged from the laboratory.

4.2.1.3 The Regenerator System

As indicated in Plate 4.2, the regenerator set-up is essentially made up of three units: a water reservoir, a boiler and a superheater.

Water is fed from the reservoir to the boiler via a glass tube. The boiler itself is a 5.0 x 10^{-3} m^3 round-bottomed pyrex glass flask with three necks, all being parallel. The water in the flask is heated using a 2 x 300 watt heating mantle. By varying the power input to the heating mantle, steam can be produced between rates of 1.6 x 10^{-5} kg.s^{-1} and 1.85 x 10^{-4} kg.s^{-1}, but only the maximum steam rate was used in all the experiments described in this Chapter. A pressure release tube, 1.045m long, is fitted to allow for pressure fluctuations in the boiler. If the pressure in the boiler becomes too high, water is forced up the tube and returned to the reservoir by PVC tubing.

The superheater is made up of an insulated copper tube, 0.80m in length and an outside diameter of 0.65×10^{-2}m. It is heated using heating tape which has a width of 0.2×10^{-1}m, is 0.3×10^{-2}m in thickness and 1.80m long. The tape, insulated with glass cloth and extruded silicone rubber, is designed to function up to a maximum temperature of 473K. The purpose of a by-pass valve positioned before the superheater section is two-fold: if steam pressure becomes too high, steam can be released by opening this valve and secondly, steam can be discharged until the adsorber is prepared for regeneration. A thermocouple is used to measure the temperature of the steam being fed to the column.

The steam flows down the vessel and desorbs the solvent taken up by the charcoal cloth. The discharged mixture is condensed and cooled by cold water as it passes through a condenser/cooler before finally being collected in a flask.

4.2.2 Analysis

4.2.2.1 During Adsorption

The solvent liquid rate and air rate to the column are fixed and by recording the atmospheric pressure and temperature for each operation, the solvent vapour concentration in the air can be evaluated.

Samples of the organic solvent-air vapour mixture are withdrawn from the system and pass through a syringe needle and plastic capillary tube before being analysed in situ by a Centronic 200 MGA mass spectrometer. The instrument can be used to find materials ranging in molecular weights from 0 - 200 and generally scanned between -5 and +5 of the molecular weight of the solvent vapour being analysed.

For each experimental run, the sensitivity of the mass spectrometer and the potential difference across the chart recorder are adjusted to obtain a suitable peak height corresponding to the amount of solvent contained in the influent vapour stream. By measuring the size of peaks recorded in analysing the effluent, the ratio of peak heights corresponding to influent and effluent solute concentrations are used in determining the quantity of organic solvent in the adsorber's exhaust stream.

4.3.2.2 During Regeneration

The condensate is collected at regular intervals during desorption. Since the solvents being investigated, toluene and perchloroethylene, are immiscible with water, the quantity of each liquid can simply be measured by pouring the condensate into a measuring cylinder.

4.2.3 Safety

Operability studies and commissioning runs were carried out before ensuring that the batch system could be operated safely. The tests undertaken during commissioning and the design of the batch set-up with regard to safety are now described.

The temperature at the surface of the glass finger was calibrated against the output voltage from the torovolt so that it was maintained well below the auto-ignition temperature of toluene. Perchloroethylene solvent, however, is non-flammable. Tests were also carried out to find the maximum rate at which the liquid solvents could be vapourized. In order to avoid any condensation of solvent vapour in the equipment, it was ensured that the liquid solvent and air rates used during the experimental runs were such that the partial pressure of the solvent vapour in the air did not exceed its vapour pressure at the operating temperature.

Since the solvent vapour concentration is regularly monitored, any undue changes in air and solvent rates are quickly noticed. The solvent vapourizing section is surrounded by a screen and in the event of an explosion, the blast is directed towards a wall and away from any persons elsewhere in the laboratory.

Pressure release devices in the form of a glass U-tube, one limb of which is connected to the air line by a 'T' piece. The vapour is vented through this tube if there should be a closed valve or high air pressure during operation.

No hazard should arise from an electrical failure since the heat input to the glass finger and syringe pump are switched off.

With regard to the regeneration set-up, initial tests were carried out to measure the maximum rate at which steam could be generated and calibrate the superheater temperature with the electrical input.

The regeneration system was investigated for any leaks prior to desorption and gaskets fitted between the various sections of the adsorber were replaced if necessary.

The water in the boiler was made up to $2.5 \times 10^{3}\ \mathrm{m}^{3}$ before each experimental run, this quantity being much greater than the steam requirements for regeneration. As stated in Section 4.2.1.3, in the event of high pressure, steam can be released through a relief valve fitted to the superheater tube and water is forced up a long narrow glass tube and returned to the water reservoir at high boiler pressures.

The size of glass condenser and cooling water rate are such to ensure complete condensation of the solvent-water vapour mixture. However, if the cooling water were to fail any vapour would be vented through a tube attached to a glass elbow which connects the condenser to the collecting flask.

4.3 Experimental Conditions and Results

Although the single and cyclic adsorption-desorption processes are treated separately in this Section, there are some general points which are common to all the operations and these are now described.

The charcoal cloth used in all the batch experiments was cut from a large roll (Reference 191 ex Nancekuke), weighed and then placed in the column. The adsorption was carried out and the system was considered to be at equilibrium when the influent and effluent concentrations of the adsorbate were similar, or when the latter remained constant. (It is noted here that it is believed that the PVC tubing fitted to the adsorber outlet absorbed some of the solvent vapours in the exhaust stream. Therefore, in many experiments, the system was considered to be at equilibrium when the outlet solvent concentration remained constant and not necessarily when the inlet and outlet adsorbate concentrations were similar.) Once the cloth adsorbent was saturated, it was removed from the column and weighed quickly in order to determine the amount of solvent adsorbed. The solvent-laden charcoal cloth was then replaced in the column and the rig set up for steam regeneration as indicated in Plate 4.2. The steam for desorption was set at the maximum rate 1.85 x 10^{-4} kg.s^{-1} in all the operations.

The relative humidity of the air varied slightly throughout each experimental run and average values are quoted in the Tables describing the experimental conditions. The temperature of the vapour stream at the inlet to and outlet from the column were recorded regularly. The temperature changes during each operation were in the order of ±2K and the adsorption temperature is taken as the average of all the temperatures recorded.

Finally, details of the experimental conditions and the adsorption regeneration and condensate recovery results for all the operations are outlined in Tables included in Appendix II.

4.3.1 Single Adsorption-Desorption

4.3.1.1 Perchloroethylene

4.3.1.1.1 Experimental Conditions

A summary of the experimental conditions in studying the adsorption of perchloroethylene vapour onto activated charcoal cloth are displayed in Table 1. In order to plot adsorption isotherms, two sets of experiments varying the volumetric concentration of the solvent vapour in the air were carried out at a constant superficial gas velocity of 0.1373 $m.s^{-1}$ (±1.75%). The effect of superficial vapour velocity on adsorption was investigated at rates of 0.040, 0.086 and 0.113 $m.s^{-1}$ at a perchloroethylene concentration of 1.215% (±2.36%).

4.3.1.1.2 Results

The adsorption results with regard to the uptake of perchloroethylene vapour onto activated charcoal cloth are reported in Table 2.

With reference to experimental numbers BP2 to BP6 inclusive, the equivalent equilibrium capacity of unit mass of charcoal cloth is shown to increase gradually from 0.732 to 0.764 $kg.kg^{-1}$ for volumetric solvent concentrations of 0.50 to 2.00% respectively. However, at a perchloroethylene concentration of 0.375% represented by BP1, the equivalent uptake of the solvent was found to be 0.753 kg per kg of charcoal cloth. It was also found from subsequent experiments indicated by BP7 to BP11, that there was a general improvement in the cloth's adsorptive capacity. The lower adsorbabilities in the earlier experiments are attributed to the cloth sections being cut from the outer layers of the large charcoal cloth roll where the adsorption of impurities from the atmosphere was more likely.

Two adsorption isotherms have therefore been drawn and are shown in Figure 4.4. It is noted that a spurious low adsorbent capacity, thought to result from the cloth becoming contaminated during handling, was determined for experiment BP9, and has not been reported on the diagram. However, the result obtained from CBP1, the first adsorption process of the cyclic operations involving the perchloroethylene vapour is included. It appears that either the Langmuir or Freundlich equations can be applied to the isotherms and a full analysis examining these theories is presented in Chapter 5.

The concentration of the perchloroethylene vapour in the air not only affects the equilibrium capacity of the cloth adsorbent, but also influences the time to attain equilibrium as well as the size and shape of the mass transfer zone as shown by the breakthrough curves in Figure 4.5.

It is seen from this Figure that in the case whereby the perchloroethylene vapour concentration was almost 2.00% by volume, the time to attain equilibrium was found to be 2820 seconds and increased almost tenfold to 27540 seconds at a concentration of 0.315%. The breakthrough point was reached after some 300 and 2500 seconds respectively. A short mass transfer zone is apparent at the high solvent concentration in contrast with a much greater zone length obtained when the perchloroethylene concentration was low at 0.375%. Two breakthrough curves are drawn for a volumetric solvent concentration of 1.00%, the adsorption being carried out on the less contaminated cloth in run BP10. In both experiments, the breakthrough point occurred after about 1600 seconds followed by a rapid increase in the perchloroethylene concentration in the exhaust. This rapid rise in adsorbate concentration was maintained until the charcoal bed was considered saturated, after 6480 seconds in Experiment Number BP4.

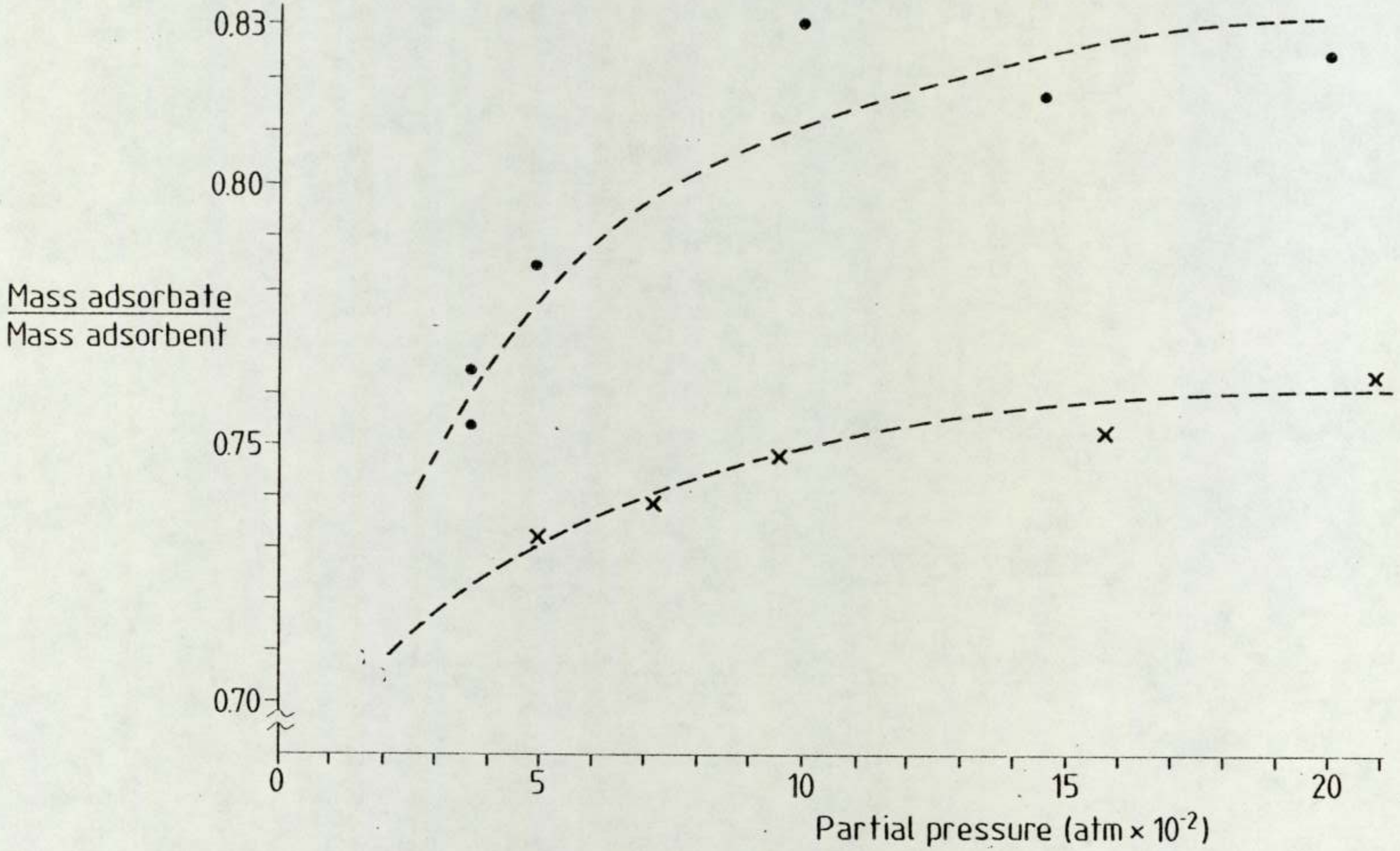


FIGURE 4.4 - ADSORPTION ISOTHERMS FOR THE UPTAKE OF PERCHLOROETHYLENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

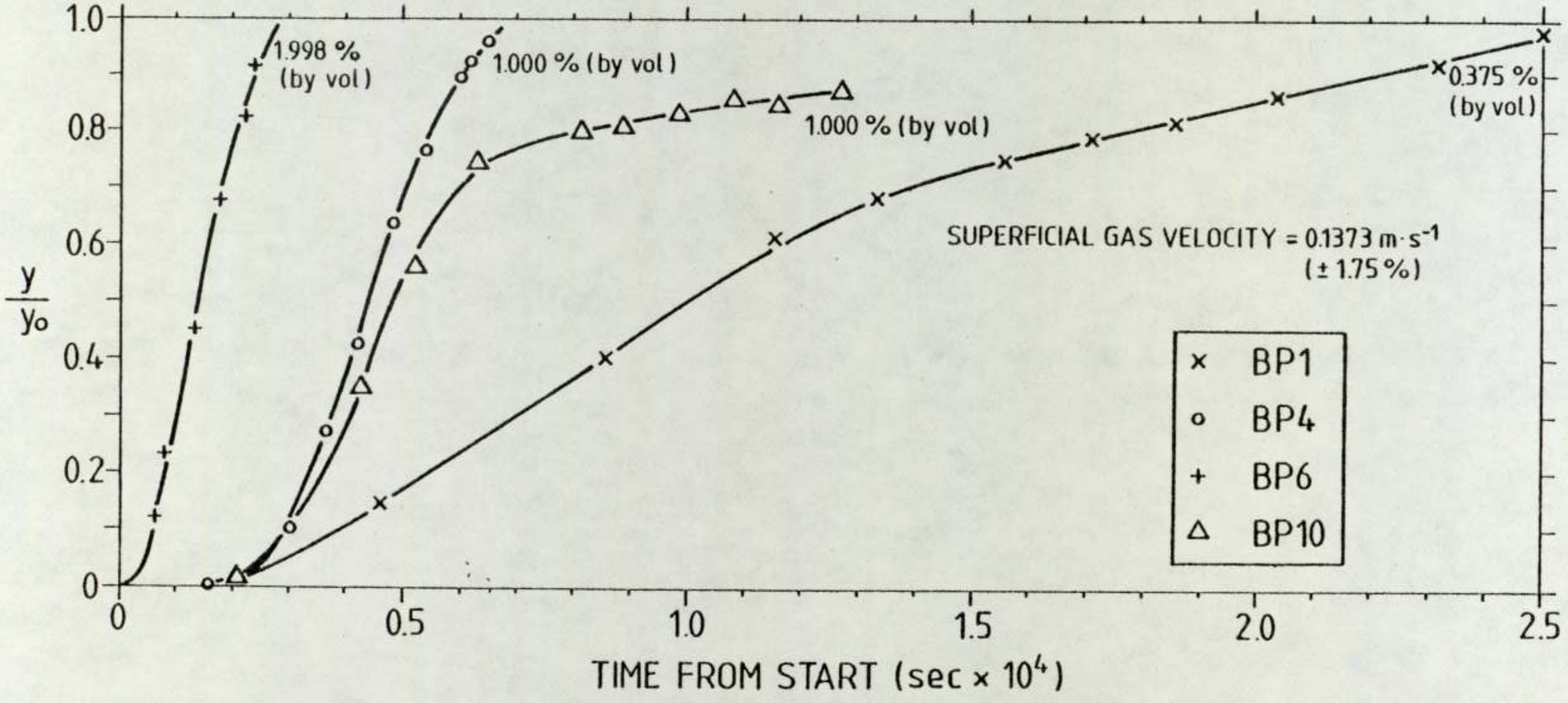


FIGURE 4.5 - EXPERIMENTAL OUTLET CONCENTRATION OF PERCHLOROETHYLENE IN AIR AGAINST TIME AT CONSTANT VELOCITY - EFFECT OF CONCENTRATION

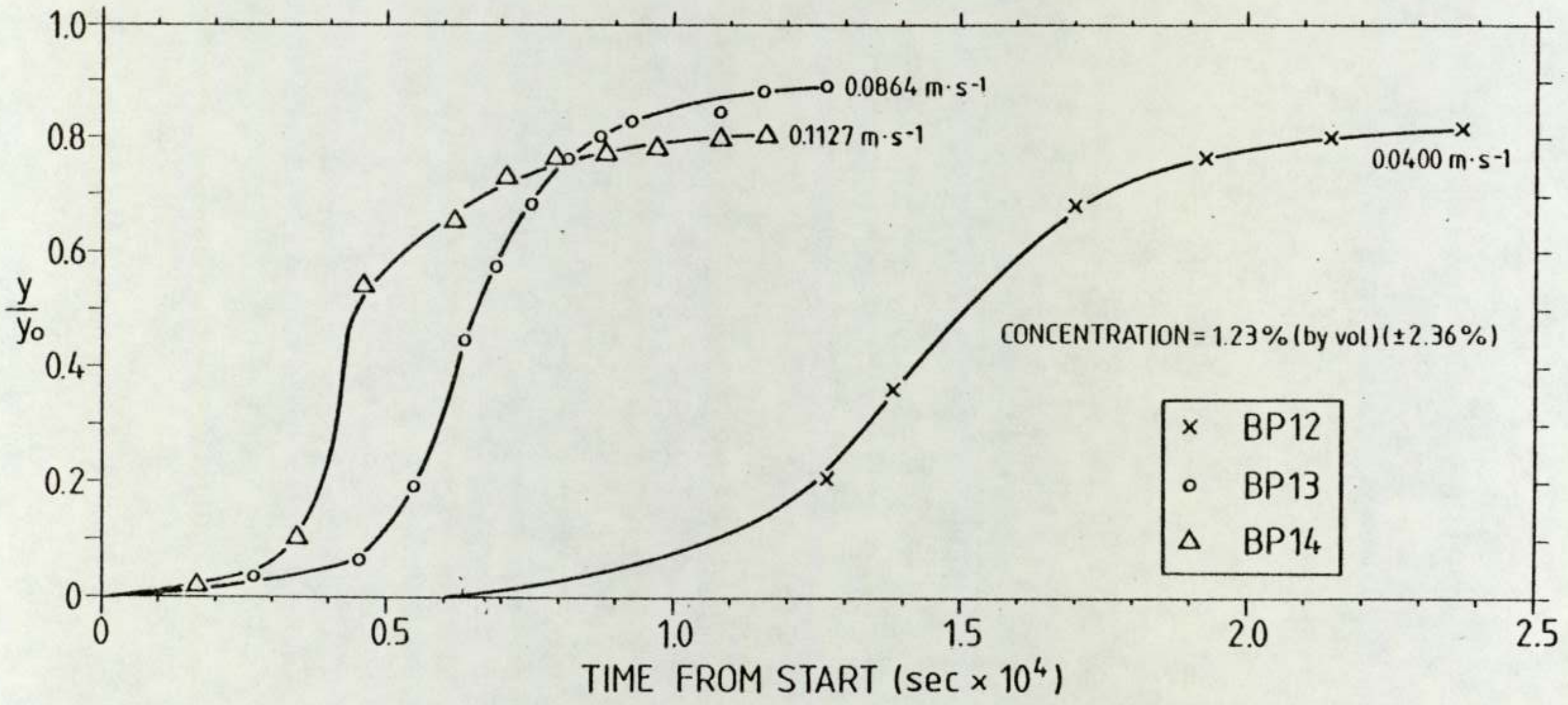


FIGURE 4.6 - EXPERIMENTAL OUTLET CONCENTRATION OF TOLUENE IN AIR AGAINST TIME AT CONSTANT CONCENTRATION - EFFECT OF VELOCITY

However, during the adsorption onto the more active charcoal cloth, the concentration of the solvent in the effluent increased quickly until it was about three-quarters that of the inlet, after which it rose slowly until 12750 seconds had elapsed when the system was considered to be at equilibrium.

The superficial gas velocity also affects the adsorptive capacity of the charcoal cloth and the adsorption rate. The results in Table 2 represented by runs BP12, BP13 and BP14 indicate that for an increasing gas velocity, the adsorptive capacity decreases. It is shown that at a perchloroethylene concentration in the air averaging 1.215%, the equivalent uptake of the solvent per unit mass of cloth adsorbent is reduced from 0.818 $kg.kg^{-1}$ at a velocity of 0.04 $m.s^{-1}$ to 0.794 $kg.kg^{-1}$ at 0.113 $m.s^{-1}$.

The breakthrough point was reached after some 1500 seconds when the superficial gas velocity was 0.0864 $m.s^{-1}$ and 0.113 $m.s^{-1}$ but increased to 6500 seconds at a velocity of 0.04 $m.s^{-1}$. The shape of the adsorption waves are shown to be roughly 'S' shaped at the three velocities examined, but the length of the mass transfer zone and the time considered for the system to have attained saturation is reduced as the gas velocity increased.

The regeneration and condensate recovery results are summarized in Tables 3 and 4 respectively. A certain amount of steam was needed during the regeneration processes to heat up the equipment, raise the adsorbent bed temperature and provide heat for desorption. In the above operations, BP1 - BP14 inclusive, steam was fed into the column for about 300 seconds amounting to some 0.055kg before any condensate was recovered. The steaming was continued for a total of 180, 360 or 540 seconds, condensate being collected and analysed every 60, 120 or 180 seconds respectively.

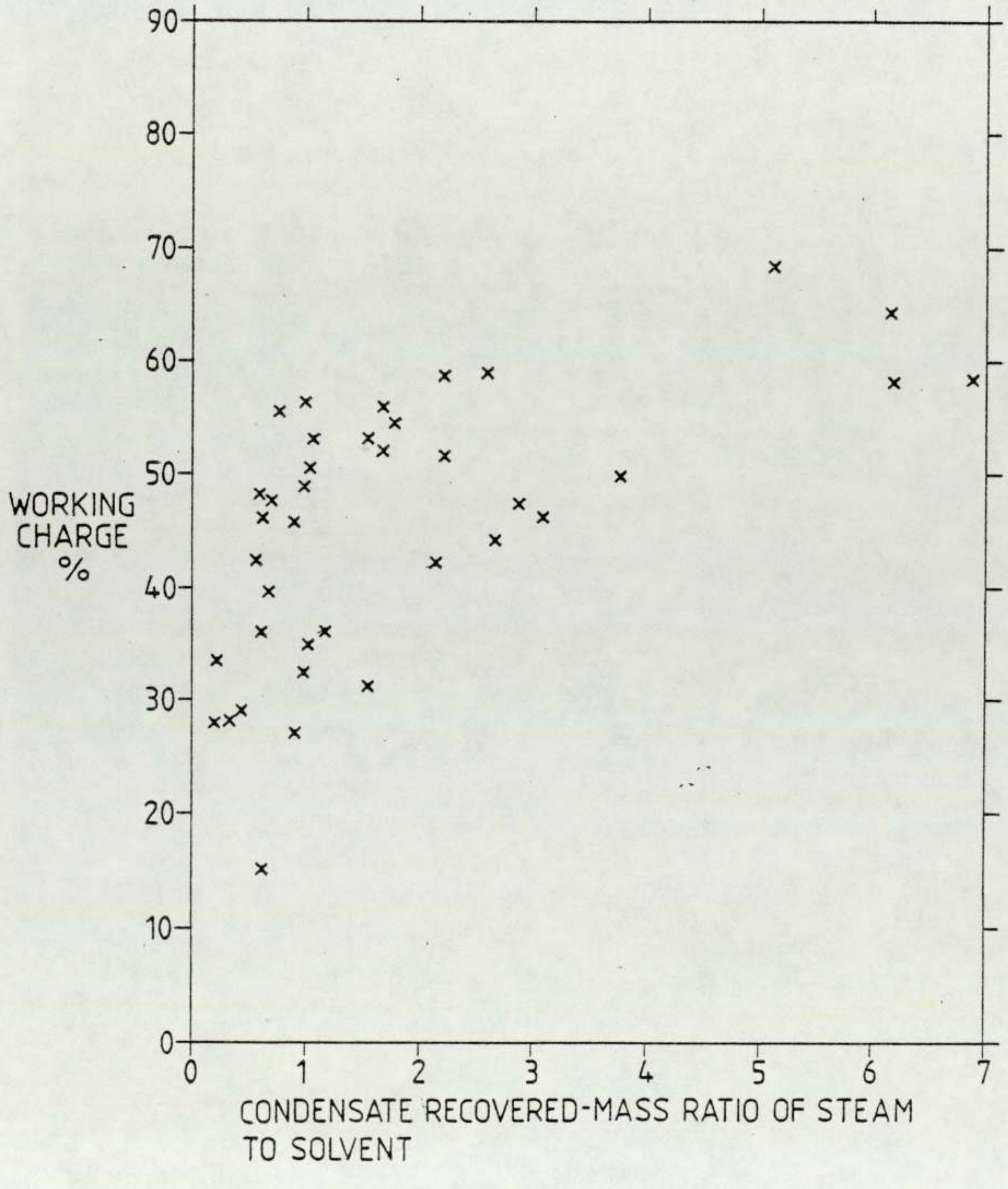


FIGURE 4.7 - PLOT OF WORKING CHARGE AGAINST MASS RATIO OF STEAM TO SOLVENT CONDENSED FOR THE RECOVERY OF PERCHLOROETHYLENE

During regeneration all the material adsorbed is not recovered, the fraction of material remaining adsorbed is known as the heel, and the working charge refers to that recovered. The working charge averaged 53.86% for all the single adsorption-desorptions involving perchloroethylene, the highest and lowest values being 64.16% and 36.30% respectively.

The overall steam-solvent mass ratios recorded were high, ranging from 7.98 down to 4.28, but a large proportion of this steam was used to heat up and was taken up by the equipment and adsorbent. It was thought that in some of the operations that steam leaks often resulting from broken gaskets led to abnormally high steam-solvent ratios. However, much lower steam-solvent ratios were obtained in the condensate as detailed in Table 4. It was during the first 180 seconds of recovering the condensate that most of the perchloroethylene was collected and it is shown in the Table that after this time, the working charge varied between 35.0 and 56.19% at steam-solvent ratios ranging from as low as 0.67 up to 1.55. Subsequently, relatively large quantities of steam were necessary to recover small volumes of solvent and the relationship between the working charge and the ratio of the mass of steam and solvent vapour condensed is shown in Figure 4.7. The scatter of points included in this Figure is attributed partly to experimental error and to the fact that although the actual mass of solvent adsorbed varied throughout the operations, the steam rate was maintained the same.

4.3.1.2 Toluene

4.3.1.2.1 Experimental Conditions

The experimental conditions under which the adsorption of toluene vapour onto activated charcoal cloth was carried out are reported in Table 5. The experimental numbers BT1 to BT7 refer to a study of the effect of the toluene concentration in the air upon adsorption equilibrium and the rate of adsorption, the volumetric concentration being increased from 0.360 to 1.921% at a constant superficial gas velocity of 0.1367 $m.s^{-1}$ (±2.41%). The concentration of the toluene vapour was unchanged at 1.172% (by volume) for operations BT8, BT9 and BT10 so that the effect of the gas velocity upon adsorption could be investigated.

4.3.1.2.2 Results

The adsorption results with regard to the uptake of toluene vapour onto activated charcoal cloth are presented in Table 6. The corresponding adsorption isotherm is shown in Figure 4.8 and, as for the adsorption of perchloroethylene vapour, it appears to conform to the theories of Langmuir or Freundlich. Since the vapour density of toluene at STP is 4.11 $kg.m^{-3}$ compared with 7.40 $kg.m^{-3}$ for perchloroethylene[90], the mass of toluene adsorbed was less than that for the chlorinated solvent vapour under similar conditions. The equivalent uptake of toluene vapour per kilogram of cloth adsorbent is shown to vary in Table 6 between 0.319 and 0.388 $kg.kg^{-1}$ at concentrations ranging from 0.360 to 1.921% (by volume). This contrasts with values increasing from 0.732 to 0.824 $kg.kg^{-1}$ for perchloroethylene solvent over a similar volumetric concentration range.

The effect of the toluene vapour concentration on the breakthrough point, the shape of the adsorption wave and the height of the mass transfer zone is presented in Figure 4.9. In comparison with the results obtained using perchloroethylene vapour, a similar general trend of a steeper adsorption wave and the time to breakthrough occurring more rapidly for increasing toluene concentration is evident. Having studied the adsorption of toluene at the relatively high inlet concentration of 1.921% (by volume) the solvent vapour in the exhaust was detected almost immediately. However, the system was considered to have reached a state of equilibrium

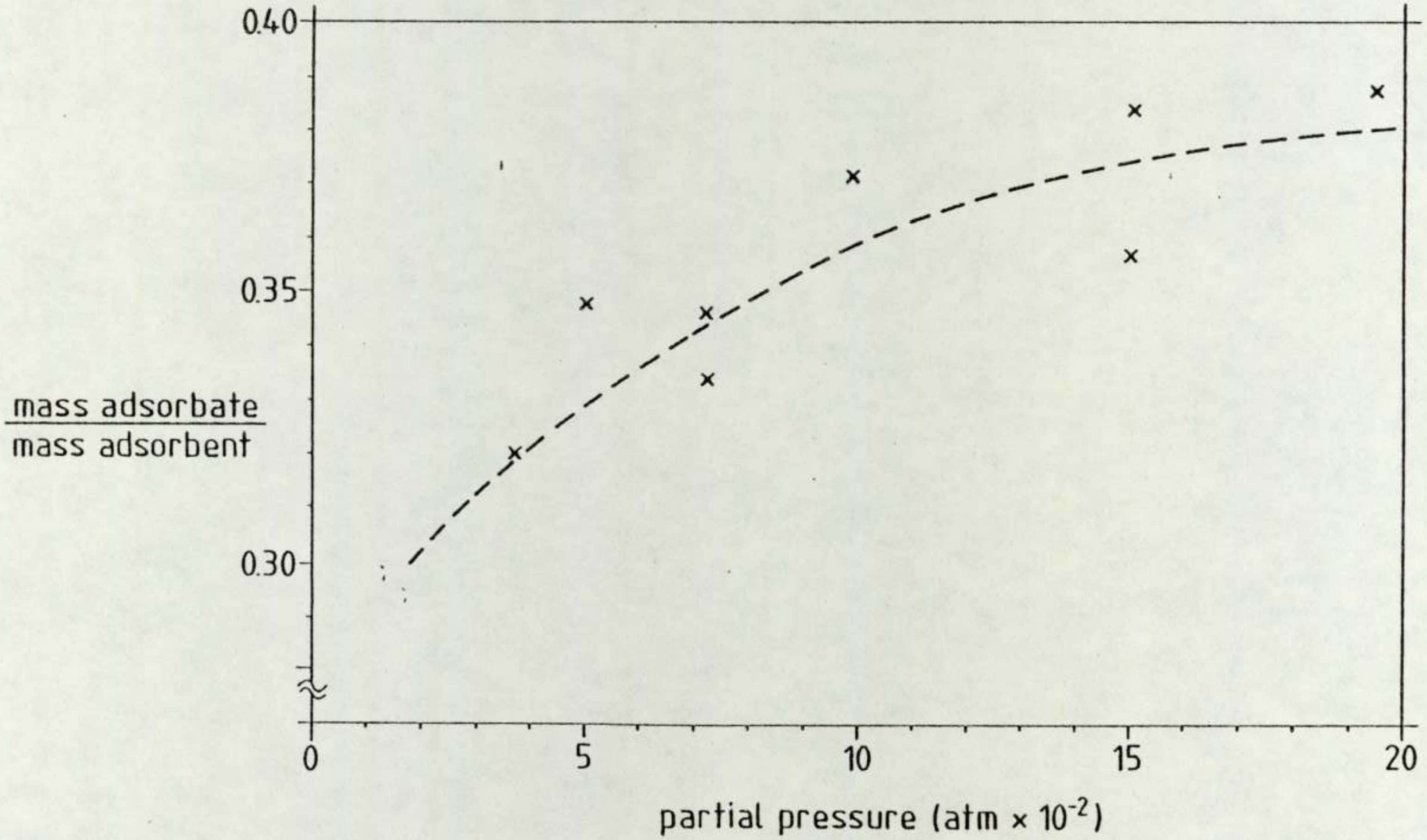


FIGURE 4.8 - ADSORPTION ISOTHERM FOR THE UPTAKE OF TOLUENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

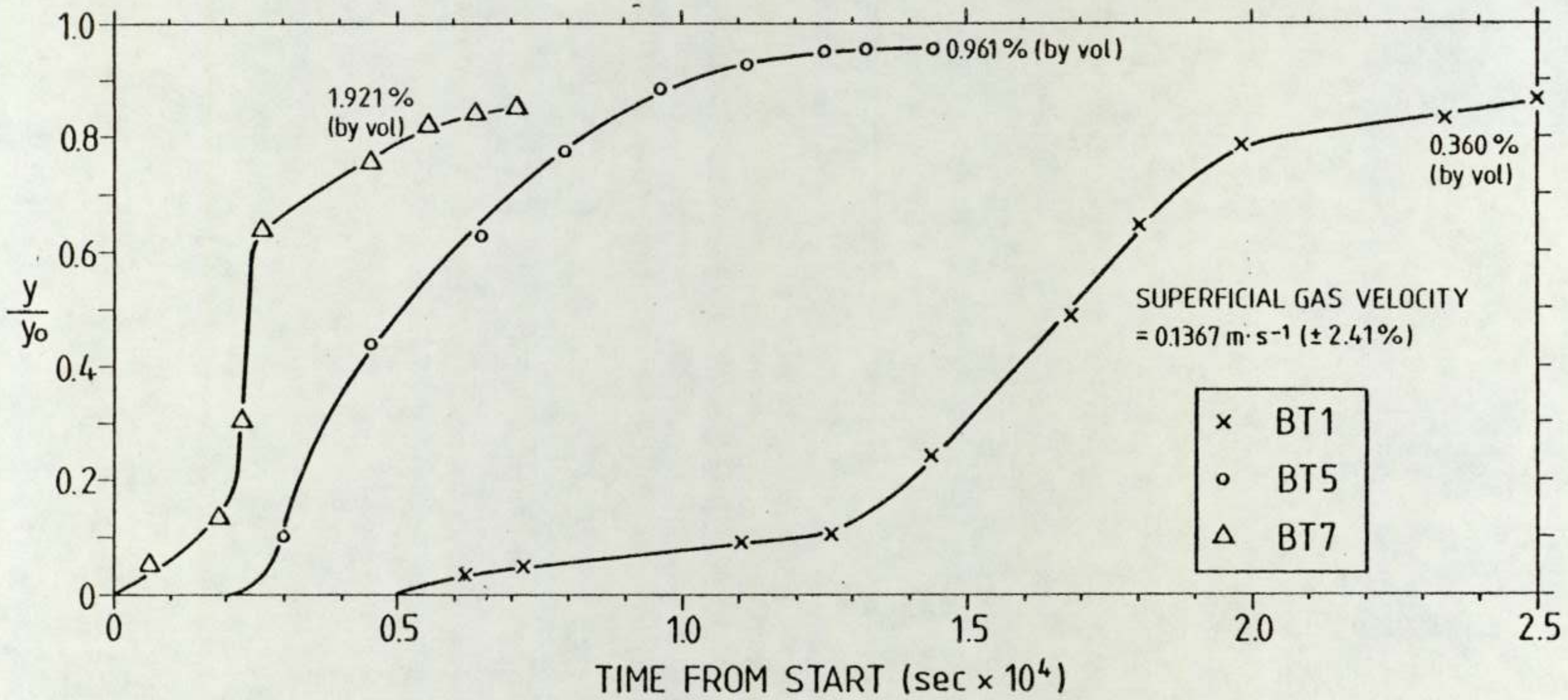


FIGURE 4.9 - EXPERIMENTAL OUTLET CONCENTRATION OF TOLUENE IN AIR AGAINST TIME AT CONSTANT VELOCITY - EFFECT OF CONCENTRATION

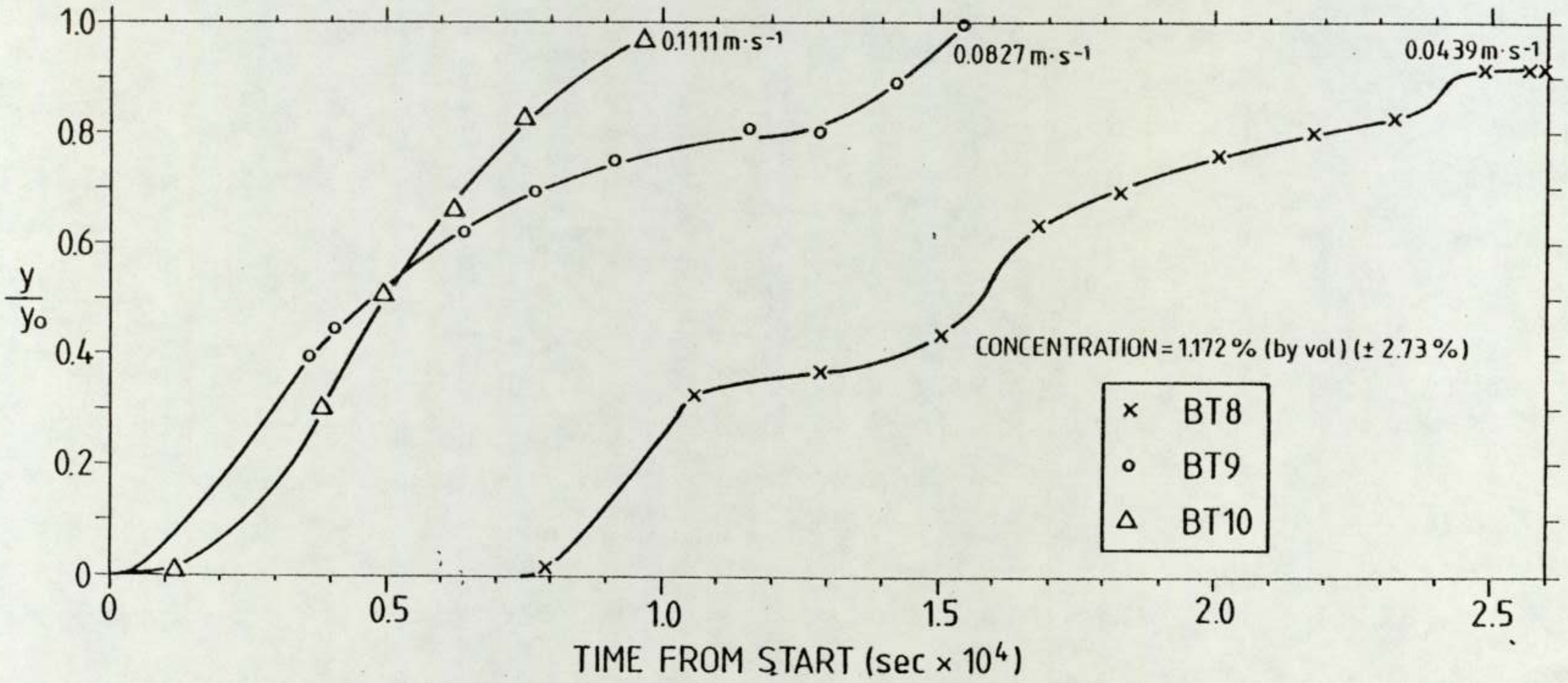


FIGURE 4.10 - EXPERIMENTAL OUTLET CONCENTRATION OF TOLUENE IN AIR AGAINST TIME AT CONSTANT CONCENTRATION - EFFECT OF VELOCITY

after 7080 seconds compared with 5460 seconds obtained for the experiment involving the uptake of the perchloroethylene vapour at a concentration of 2.00% (by volume). It can also be noticed from Figure 4.8 that at the relatively low and medium volumetric concentrations of 0.360% and 0.961%, the respective breakthrough point was found to have occurred after 2500 and 5000 seconds and the time to saturation after 12750 and 27540 seconds. During the adsorption of the chlorinated solvent at similar concentrations, the breakthrough point was reached more rapidly, but there was relatively little difference between their saturation times.

The equivalent uptake of the toluene per unit mass of charcoal cloth was found to be 0.360, 0.370 and 0.351 kg.kg at a concentration of 1.172% (by volume) at the relatively low, medium and high gas velocities of 0.0439, 0.0827 and 0.1111 $m.s^{-1}$ respectively. Therefore, the capacity of the cloth adsorbent was not found to be highest at the lowest gas velocity which was the case for the adsorption of perchloroethylene. It is believed, however, that the cloth's capacity would have been greatest at the lowest gas velocity examined if it were not for the high relative humidity of 59%.

The adsorption rate is also affected by the superficial gas velocity and the relationship between the concentration of the exhaust gases from the adsorber at the three different velocities are shown in Figure 4.10. It can be observed from this Figure that during experiments BT8 and BT9 corresponding to gas velocities of 0.0439 and 0.0827 $m.s^{-1}$ respectively, the rate of increase in the outlet toluene concentration in the air varies with time compared with a gradual rise obtained at a velocity of 0.1111 $m.s^{-1}$. The time to breakthrough was 6100 seconds at the low velocity falling to 400 seconds at the medium velocity, but increasing to 1250 seconds at the high velocity. Although the breakthrough point occurred earlier at the medium velocity, a state of equilibrium had not been reached until some 15480 seconds had elapsed since commencing the adsorption process. Saturation of the adsorbent cloth with toluene was considered after 26340 and 9540 seconds at the gas velocities of 0.0439 and 0.1111 m.s^{-1} respectively. In contrasting the adsorption results involving the toluene and perchloroethylene vapours, it was found that the time to attain equilibrium was longer for the toluene at the low and medium velocities, but the breakthrough point was more rapid in all the three velocities examined.

The regeneration and condensate recovery results are outlined in Tables 7 and 8 respectively. As was the case for operations BP1 to BP14 involving the recovery of perchloroethylene solvent, steam was fed into the column for an average of 281 seconds before any condensate was recovered.

The overall steam-solvent mass ratio was again high, ranging from 9.31 up to 15.23 and a mean value of 11.81. However, the steam solvent mass ratios measured in the condensate were much lower. It was found that the greatest majority of toluene was, as for perchloroethylene, recovered after 180 seconds, and in analysing the condensate, the ratios of the mass of steam to the solvent vapour condensed differed between 1.57 and 2.53. These values are higher than the corresponding results obtained in recovering the chlorinated solvent, but the liquid density of perchloroethylene is 1623 kg.m^{-3}, almost twice that of toluene which is 866 kg.m^{-3}[90].

The working charge varied between 37.25 and 59.26% after 180 seconds and are similar to the range of working charges from 35.00 up to 56.12% determined for the perchloroethylene recovery. The working charge averaged 54.26% for all the single adsorption-desorption experiments with regard to toluene ranging from a remarkably high percentage of 82.06 down to 37.95%.

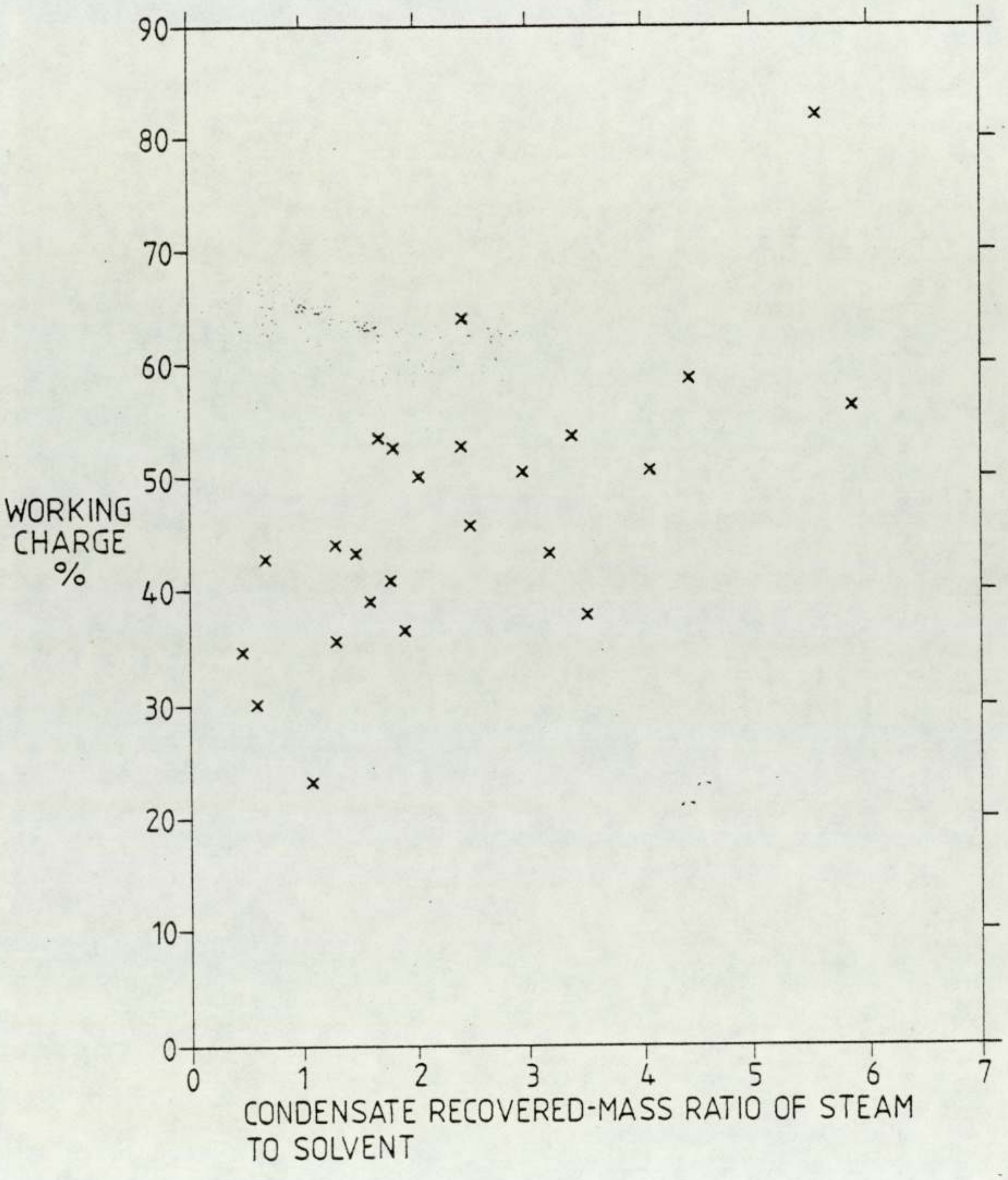


FIGURE 4.11 - PLOT OF WORKING CHARGE AGAINST MASS RATE OF STEAM TO SOLVENT CONDENSED FOR THE RECOVERY OF TOLUENE

A plot of the working charge against the mass of steam to toluene vapour condensed is included in Figure 4.11. It is again evident that there is a large scatter of points and beyond a steam-solvent ratio of 3.0, the increase in working charge is very small and a similar phenomenum is noticed for the recovery of perchloroethylene.

4.3.2 Cyclic Adsorption-Desorption

4.3.2.1 Experimental Conditions

Details of the experimental conditions involving the cyclic adsorption-desorption operations for both perchloroethylene and toluene vapours are presented in Table 9.

Four cyclic runs were carried out using perchloroethylene, the volumetric concentration of the solvent being 1.50%. The mean relative humidity of the air stream was 47.2% and the adsorption temperature averaged 295.25K.

The conditions under which the five cyclic adsorption-desorption processes involving toluene vapour were similar to those using the chlorinated solvent; the mean temperature at which the uptake of toluene took place being 296.25K and the relative air humidity, 48%. The concentration of the toluene vapour in the air was 1.441% (by volume).

4.3.2.2 Results

The adsorption results with respect to the removal of perchloroethylene and toluene vapours are summarized in Table 10.

It is noted here that at equilibrium, the adsorbate in the first of the cyclic operations, CBP1 and CBT1, consists solely of solvent vapour, but in the subsequent processes, some water as well as solvent will remain adsorbed after drying the cloth following regeneration. The proportion of water and solvent adsorbed is not known, but the ratio of the weight of solvent taken up during adsorption to the weight of virgin cloth has been computed and the results are shown in the Table. The weight of toluene taken up by the fresh cloth was 0.0165kg compared with 0.0369kg for perchloroethylene. The mass ratio of adsorbate to adsorbent was 0.357 and 0.816 for toluene and perchloroethylene respectively. Once desorption was completed and the cloth dried, the ratio of the mass of solvent vapour adsorbed to the original mass of the activated charcoal cloth is reduced as the quantity of material adsorbed at the start of an adsorption run is increased. This is clearly shown in Table 10.

The adsorption waves for the uptake of perchloroethylene vapour from air are shown in Figure 4.12 and in Figure 4.13 for the first four cyclic runs involving toluene.

With regard to the chlorinated solvent, the breakthrough point for the first adsorption process occurred after about 2000 seconds compared with times between 500 and 1000 seconds in the subsequent operations, CBP2, CBP3 and CBP4. The time for the system to attain equilibrium decreased with the increasing number of cycles from 12540 seconds in run CBP1 down to 5040 seconds in run CBP4. Such a trend, however, is not evident with respect to the uptake of toluene vapour.

It can be seen from Figure 4.13 that the breakpoint occurs after about 1000 seconds in the first four operations. The time for the cloth to become saturated with solvent was longest in the first run at 10200 seconds after which there were fluctuations in this time with the increasing number of cyclic operations.

The regeneration results for all the cyclic adsorption-desorption operations are presented in Table 11. Condensate was recovered for a total of 360 seconds in each of these experiments and details of the condensate recovery results are shown in Table 12.

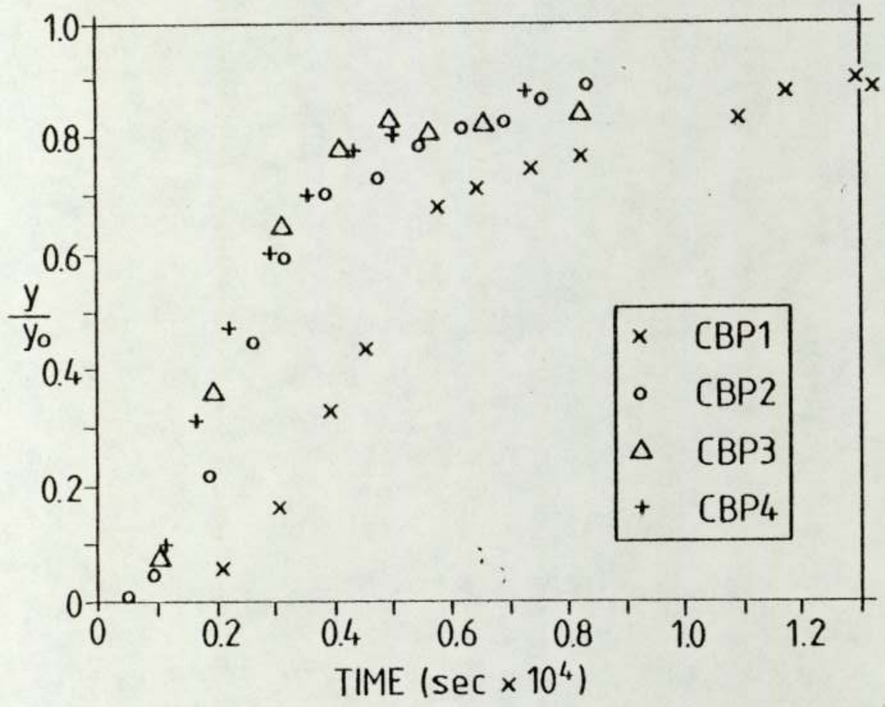


FIGURE 4.12 - EXPERIMENTAL OUTLET CONCENTRATION OF PERCHLOROETHYLENE IN AIR FOR CYCLIC ADSORPTION - DESORPTION OPERATIONS

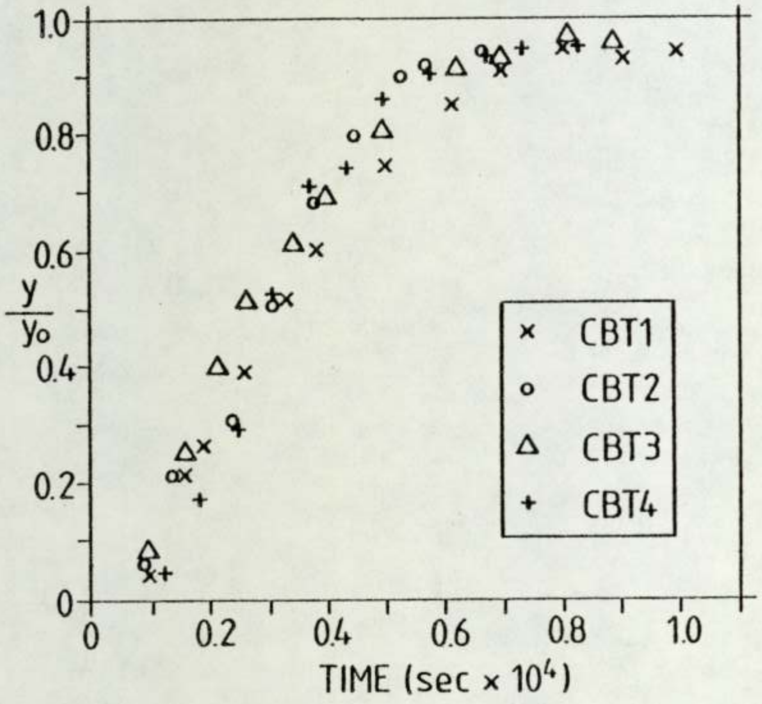


FIGURE 4.13 - EXPERIMENTAL OUTLET CONCENTRATION OF TOLUENE IN AIR FOR CYCLIC ADSORPTION - DESORPTION OPERATIONS

Steam was fed into the column for an average of 260 seconds before any condensate was collected. The weight of perchloroethylene solvent removed from the air during the adsorption period was greater than the solvent condensed during regeneration, but in the case of toluene, the amount of solvent collected was the greater in operations CBT2, CBT4 and CBT5. It is stated above that after the charcoal cloth has been regenerated and dried, some solvent and water remains adsorbed. This evidence, therefore, seems to suggest that for a particular operation, either toluene not previously desorbed is removed in addition to all that taken up during the adsorption period or the adsorbed water molecules are being replaced by the toluene molecules.

Since the actual amount of solvent remaining adsorbed after regeneration is unknown, the working charge could only be determined for the first of each of the series of operations. This was found to be 23.26% in the case of toluene and 56.73% for perchloroethylene. However, for the subsequent cyclic processes, the percentage of solvent recovered in relation to the mass of virgin cloth has been calculated. This value fluctuated for both solvents and a mean value of 45.83% was obtained for perchloroethylene and 24.2% for toluene.

It is also evident from Table 12 that most of the recoverable solvent is desorbed during the first four minutes the condensate was collected. The steam-solvent mass ratio after the condensate recovery period, increased with the increasing number of adsorption-desorption processes from 1.92 to 2.91 for the chlorinated solvent and from 4.09 up to 5.99 in the case of toluene.

4.4 General Discussion

This Section first discusses the optimum conditions for the batchwise adsorption of perchloroethylene and toluene vapours using the evidence obtained from the operations described above. A comparison is then made between the potential of activated charcoal cloth and granular charcoal with regard to the batchwise adsorption and recovery of the solvent vapours. Finally, a discussion is made between several possible types of adsorber systems incorporating charcoal cloth.

It is evident from the adsorption results that, for a given superficial gas velocity, the higher the solvent vapour concentration in the air, the greater the equilibrium capacity of the cloth. However, since on solvent recovery plants the adsorption is only carried out until the breakthrough point has been attained, the uptake of solvent by the cloth at breakthrough is more significant. It is seen from Equation 3.17 that the longer the time to breakthrough, t_B, the shorter the length of the unused bed, L. Therefore, the size of the mass transfer zone is critical. Consequently it may not necessarily be favourable to operate at the highest possible adsorption concentration.

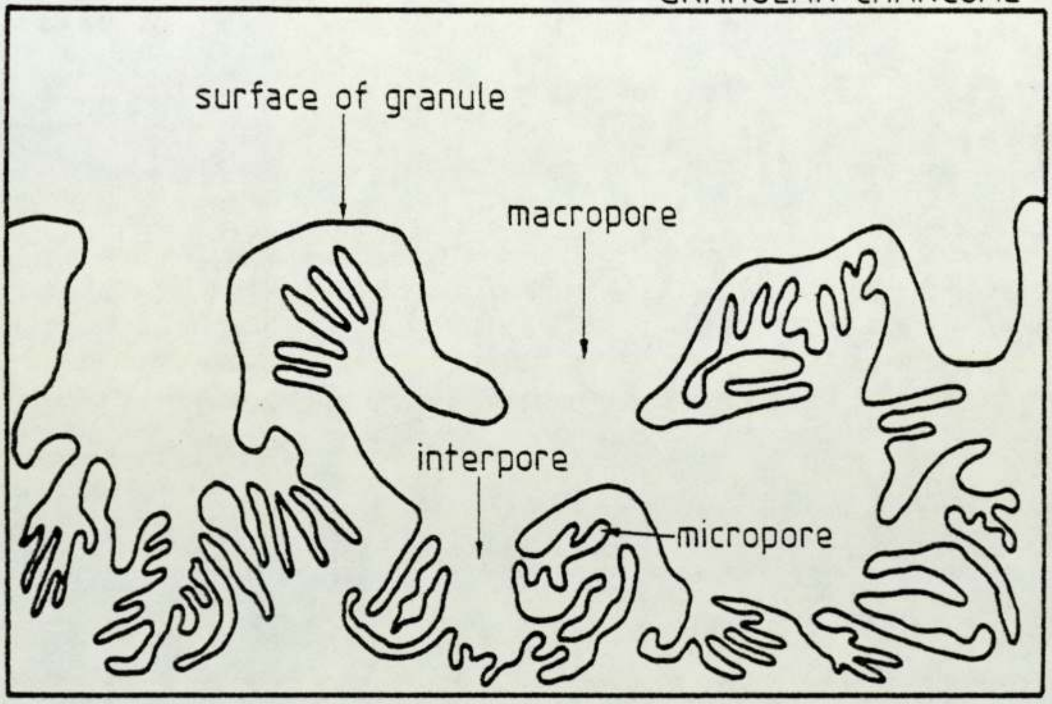
$$L = D - \left(\frac{G_s \, t_B \, (Y - Y_o^*)}{\rho_s (W_e - W_o)} \right) \qquad 3.17$$

At a given superficial gas velocity, it was found that the greatest breakthrough time occurred at the lowest adsorbate concentration and that for a set solvent concentration, the time to breakthrough also increased with a decreasing gas velocity. It follows that at low gas velocities, the residence time during which the solvent vapour remains in the vicinity of the charcoal adsorbent is relatively high. Consequently, the opportunity for adsorption is enhanced resulting in comparatively high charcoal cloth capacity. Bearing this in mind and that the breakthrough time was highest for the lowest velocity of the gas stream, it appears that the optimum adsorption operating conditions for the above system for a given temperature and humidity occur at the lowest adsorbate concentration and at the lowest superficial gas velocity.

In his thesis, Wilkins[32], commenting on work carried out by Bailey et al[91], states that charcoal cloth is made up entirely of micropores of less than 5nm, the majority of them being slit-shaped and short in length. Granular and powdered charcoals, however, are shown to consist of macro, transitional and micropores thus conforming to the adsorbent structures classified by Dubinin[82]. A comparison between the porous structures of granular charcoal and charcoal cloth is represented diagrammatically in Figure 4.14. Table 3.1 (Chapter 3) clearly shows that the bulk of the surface area of activated carbons are within their micropores which seems to suggest the higher values of surface area obtained for the charcoal cloth. Bearing this in mind, therefore, it would be expected that under similar conditions, the capacity of charcoal cloth would be greater than the nutshell charcoal. In fact, as outlined in Chapter 2, results from experiments carried out at Porton Down involving the dynamic adsorption of various solvents onto charcoal cloth and granular charcoal clearly show that the adsorptive capacity of the cloth is superior to that of granular charcoal, particularly under humid conditions. The relative increase in the cloth's performance was dependent on the type of adsorbate. With reference to Table 2.5 (Chapter 2), the uptake of carbon tetrachloride was highest and chlorobenzene lowest under both dry conditions and at 80% RH. Under dry conditions, the increase in the cloth's capacity over granular charcoal was found to range from 106.7% down to 67.2% and from 179.4% up to 817.6% at 80% humidity.

It is believed that the difference between the relative increases in the cloth's performance are attributed to the size and shape of its pores and its porous structure which are, in this case, more suitable for the size, shape and orientation of the carbon tetrachloride molecules. It could possibly be said that such properties of granular charcoal are more desirable than charcoal cloth for the uptake of water molecules which may explain the large fall in the nutshell charcoal's adsorptive capacity for

GRANULAR CHARCOAL



CHARCOAL CLOTH

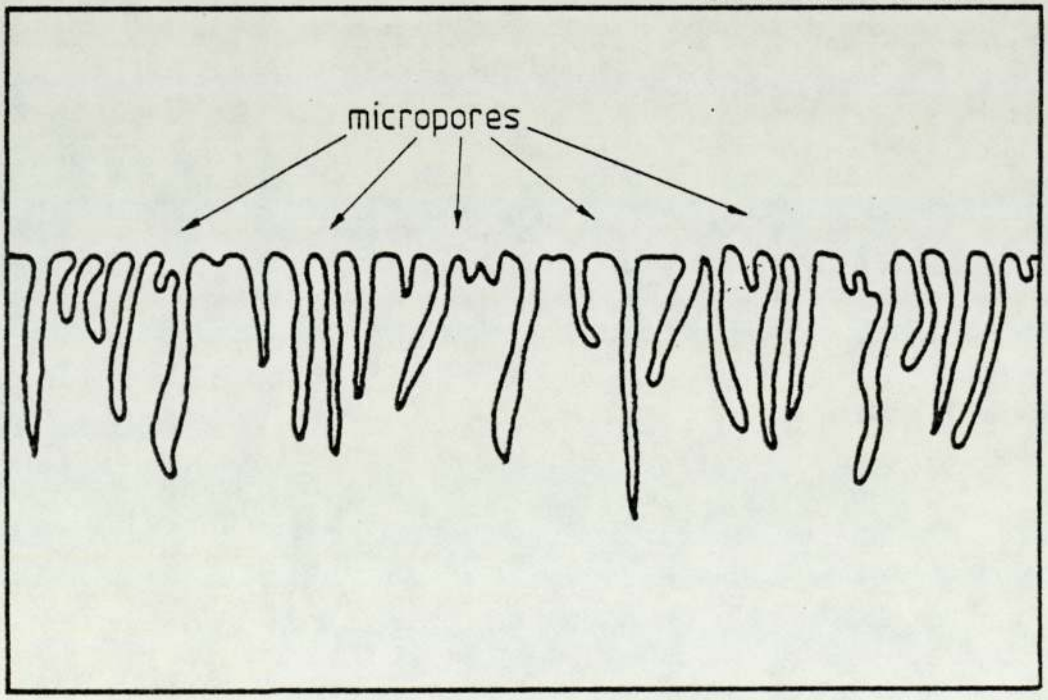


FIGURE 4.14 - COMPARISON BETWEEN THE POROUS STRUCTURES OF GRANULAR CHARCOAL AND CHARCOAL CLOTH

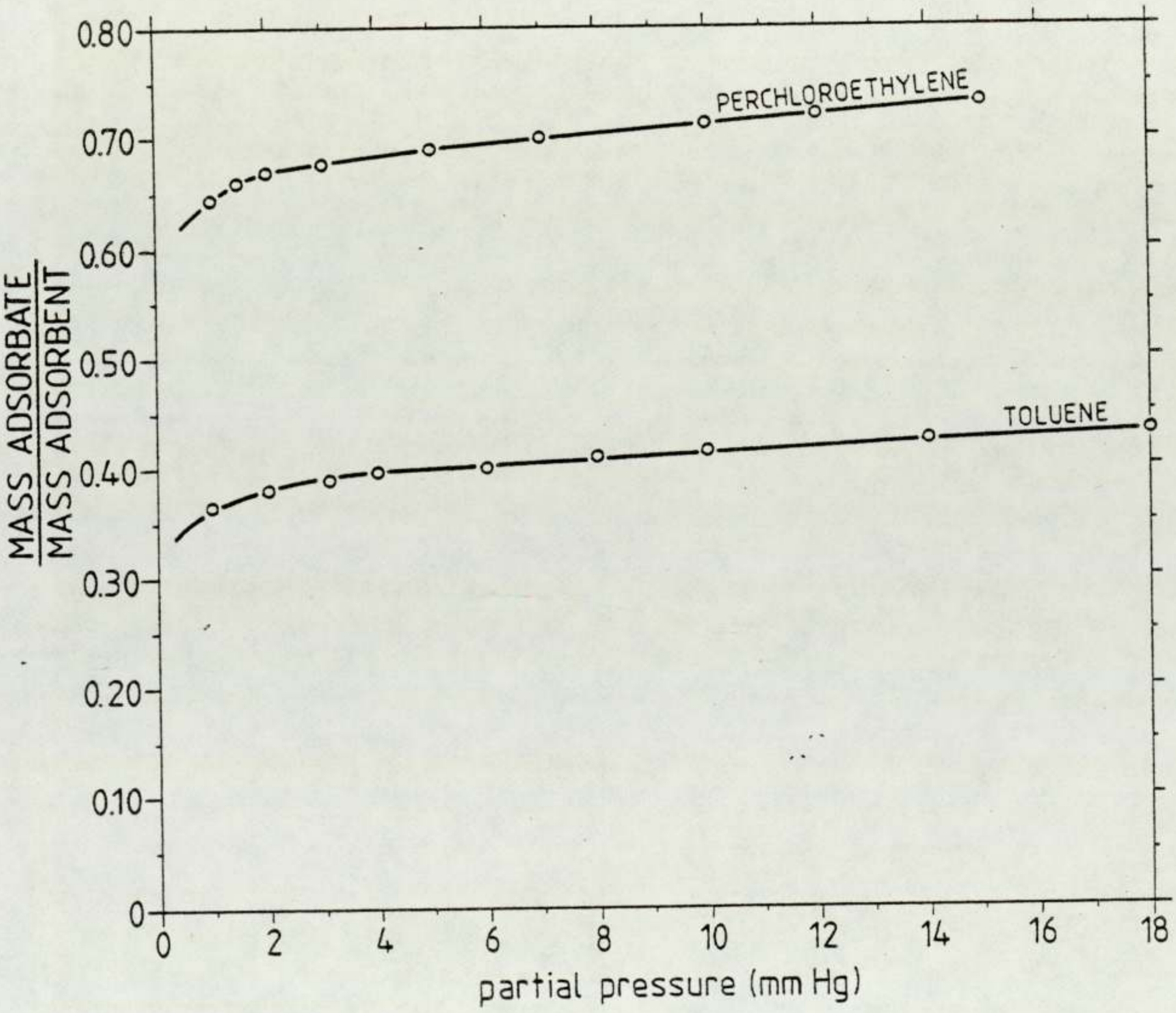


FIGURE 4.15 - ADSORPTION ISOTHERMS FOR THE UPTAKE OF PERCHLOROETHYLENE AND TOLUENE VAPOURS ONTO NUTSHELL CHARCOAL

solvent vapours under humid conditions. Since the adsorbability of the charcoal cloth also deteriorates at high humidities, it is suggested the uptake of solvents on solvent recovery plants should be carried out under dry conditions.

Dynamic adsorption isotherms involving perchloroethylene vapours, toluene vapoursand nutshell charcoal have been determined by Wildman[92] using a dynamic adsorption method developed by Davies[93], and are shown in Figure 4.15. It is not possible to derive any meaningful conclusions in comparing these results with those obtained for charcoal cloth presented in Figures 4.4 and 4.8, since the techniques and conditions under which the dynamic adsorption processes took place were completely different. However, it is clearly seen that the isotherms are similar for the adsorption onto both charcoal cloth and nutshell charcoal.

The bulk density of granular charcoal is in the order of 6100 kg.m^{-3} which is some 2000 times greater than that of activated charcoal cloth. Therefore, in considering identical masses, the volume necessary to hold the granular charcoal would be much less. As shown in Appendix 1, if it were possible to pack the charcoal cloth such that it had a bulk density of 300 kg.m^{-3}, the pressure drop per unit bed length would be ten times greater than if using the smallest available size of granular charcoal.

It is outlined in Chapter 2 that the greatest percentage of running costs in operating a solvent recovery plant is for the steam used in regeneration. Steam costs are also ever increasing, therefore, any reduction in steam requirements would be beneficial.

It is shown in Table 2.6 that with regard to nutshell charcoal, the cyclic adsorption efficiencies for Arklone P (perchloroethylene) and toluene are 20.8% and 9.6% respectively. The corresponding values obtained for the uptake of the chlorinated solvent and toluene onto charcoal cloth averaged 45.8 and 24.2% respectively. The data of Hill[47,48] shown in Table 2.6 refers to adsorption being carried out until the breakthrough point had been reached in comparison with saturated adsorption capacities measured for solvent uptake onto activated charcoal cloth.

Since it was not possible to obtain data for the ratio of steam to solvent vapour condensed and collected with regard to desorption from coconut shell-based charcoals, a useful comparison of the relationships between the working charge and steam solvent ratios for both granular and cloth charcoals cannot be made.

However, the quantities and efficiencies by which the solvents were recovered from charcoal cloth are regarded as being very high. The reason for the favourable results is thought to stem from the structure of activated charcoal cloth. It is believed that the resistances to heat and mass transfer are minimal since the adsorbed molecules are held in short micropores which are directly exposed to the outer surface of the adsorbent medium. This contrasts with the complex network of macro-, inter- and micropores inherent in granular charcoal adsorbents.

The cost of granular charcoal ex Sutcliffe Speakman is £1.40 per kilogram[92]. The cost of charcoal cloth is £16.00 per square metre, equivalent to £145.00 per kilogram[94]. Since, at the present time charcoal cloth is manufactured on a limited scale, it is anticipated that there will be a significant fall in its price with increased production.

The capital expenditure necessary for an adsorber would be much higher in the case of activated charcoal cloth because of its relatively low bulk density. If the charcoal cloth were wound around an annulus or packed in discs so as to give a high bulk density, the pressure drop would not be acceptable.

Although the capacity of unit mass of charcoal cloth is greater than nutshell charcoal with regard to the uptake of solvent vapours, and that the regeneration results appear to be very favourable, it cannot be assumed that a solvent recovery system using charcoal cloth is the more acceptable. The fact that the cost of the activated charcoal cloth adsorbent, at present, is very high and that the solvent uptake per unit volume of cloth is much lower than for granular charcoals supports this.

However, charcoal cloth can be used in a continuous arrangement whereby an endless band of the material is continually driven from an adsorber unit to a steam regenerator and then returned to the adsorbing section. A description of such a set-up is described in Chapter 6 along with details of results and recommendations obtained from experiments carried out on the equipment.

Additionally, charcoal cloth has market potential in several other fields and these are highlighted in Chapter 7.

CHAPTER 5

THEORETICAL DEVELOPMENT AND MATHEMATICAL MODELLING OF THE BATCH SYSTEM

5 THEORETICAL DEVELOPMENT AND MATHEMATICAL MODELLING OF THE BATCH SYSTEM

5.1 Introduction

The objective of the model described here is to predict the amount of charcoal cloth required for given operating conditions in a solvent recovery process.

Although the derived equations describing the adsorption isotherms, adsorption waves and rate of mass transfer expressed in terms of the length of unused bed at breakthrough are peculiar to the operations carried out on the batch rig, it is intended that the theoretical development will provide a basic framework for the adsorption of vapours in general.

5.2 Adsorption Isotherms

As outlined in Section 3.1.1.1, there are three commonly used mathematical expressions used to describe vapour adsorption equilibria as shown by the Langmuir, Brunauer, Emmet and Teller and Freundlich isotherms. The isotherms illustrated in Figures 4.4 and 4.8 corresponding respectively to the equilibrium data with regard to the uptake of perchloroethylene and toluene vapours onto activated charcoal cloth are similar to those described by Langmuir and Freundlich.

Summaries of the data used to test the application of the Langmuir and Freundlich isotherms for the adsorption of the two solvent vapours onto activated charcoal cloth have been tabulated and are included in Appendix III. Graphical plots of $1/V$ against $1/P$ and ln.V against ln.P for both the chlorinated solvent and toluene vapour adsorptive systems are illustrated by Figures 5.1 and 5.2. A linear regression analysis and the deviation from the perfect fit straight line have been undertaken on each set of the data and the results are reported in Table 5.1 below.

Solvent	Inclusive Run Range	Plot	Slope of Straight Line	Intercept on Ordinate	Deviation from Perfect Fit (Perfect Fit = 1)
			$1/_{k}V_m$	$1/V_m$	
Perchloroethylene	BP2-BP6	$1/_{v}$ v $1/_{p}$	0.002593	9.628	0.9544
Perchloroethylene	BP7-BP11, CBP1	"	0.003689	8.730	0.9496
Toluene	BT1-BT7, CBT1	"	0.008300	10.590	0.8770
			$1/_{n}$	ln.k'	
Perchloroethylene	BP2-BP6	lnV v lnp	0.029550	-2.158	0.9810
Perchloroethylene	BP7-BP11, CBP1	"	0.050600	-1.984	0.8897
Toluene	BT1-BT7, CBT1	"	0.086600	-2.046	0.8213

Table 5.1 - Results of the linear regression and deviation from the perfect fit straight line for testing the application of Langmuir and Freundlich isotherms regarding the adsorption of perchloroethylene and toluene vapours onto activated charcoal cloth

It can be seen from Table 5.1 that in general, the deviations from the perfect fit are similar for the application of both the Langmuir and Freundlich isotherms. Since Langmuir's theory suggests that the adsorbed layer is monomolecular, it appears that both the perchloroethylene and toluene molecules adhere to the surfaces of the charcoal cloth in single layers. The data also infers that the exponential equation of Freundlich can also be applied, particularly with regard to the adsorption of perchloroethylene represented by run numbers BP2 - BP6 inclusive.

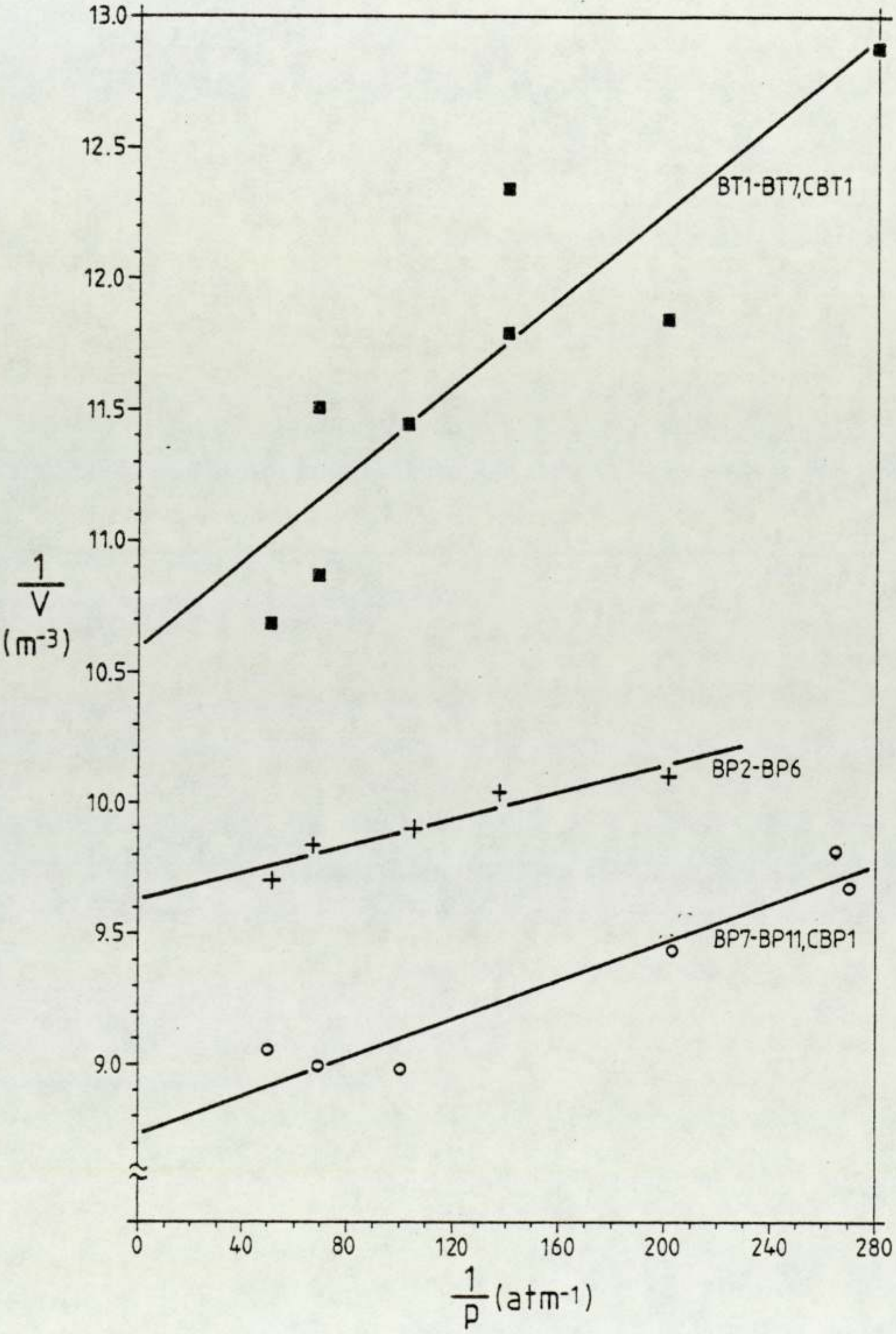


FIGURE 5.1 - PLOT OF $\frac{1}{v}$ AGAINST $\frac{1}{p}$

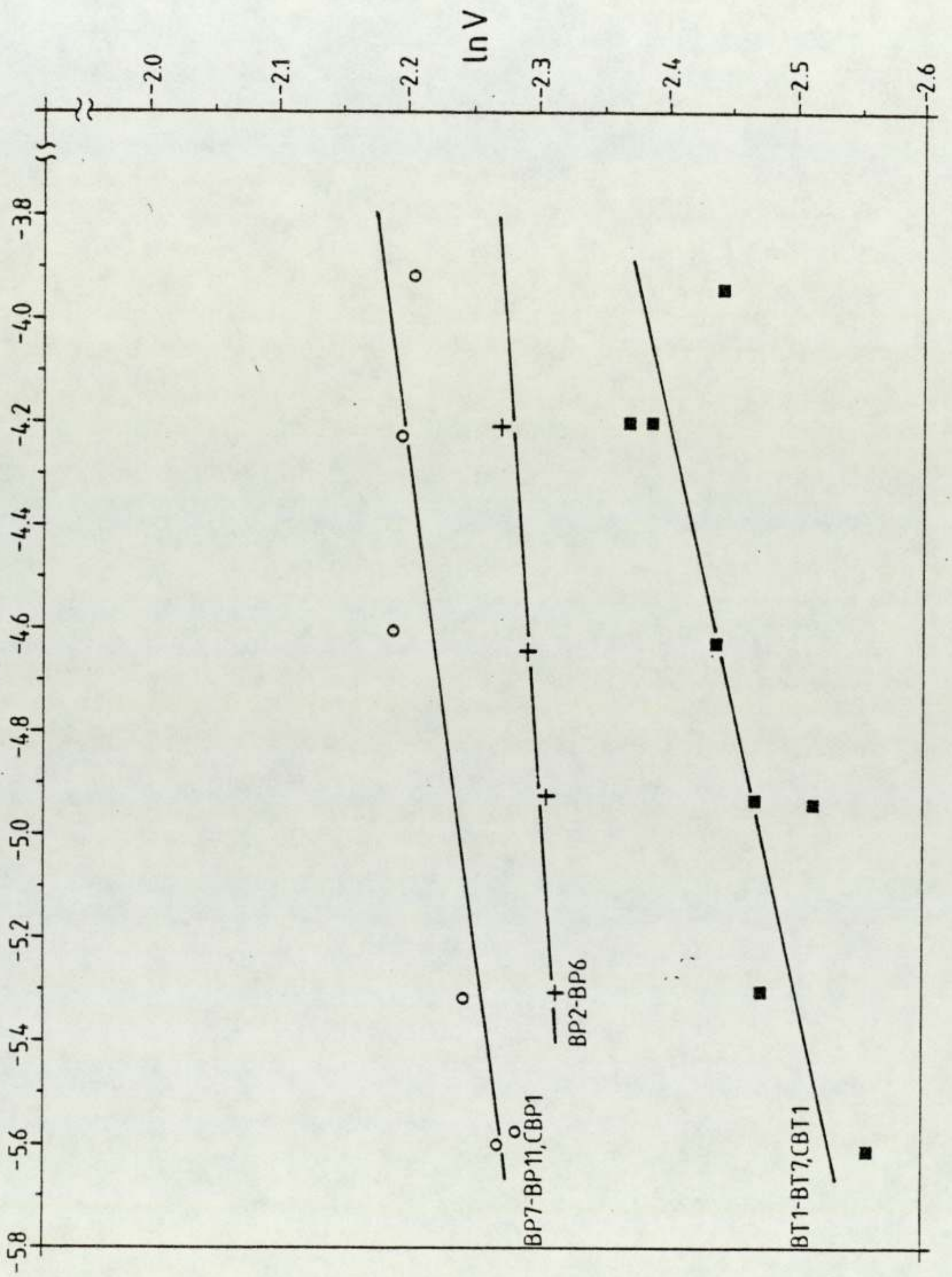


FIGURE 5.2 - PLOT OF ln v AGAINST ln p

5.3 Adsorption Waves

Adsorption waves have been constructed for particular batch operations and these are shown in Figures 4.5, 4.6, 4.9 and 4.10. Doulah[95] suggested that such curves can be represented by the general expression

$$\ln\left(1 - \frac{y}{y_o}\right) = -\left(\frac{t}{\gamma}\right)^{\beta} \qquad 5.1$$

where
y_o = volumetric solvent vapour concentration at inlet to adsorber
y = volumetric solvent vapour concentration at outlet at time, t
β = constant
γ = constant

Since ln(1) when evaluated becomes zero, Equation 5.4 can be expanded:

$$\ln(1) - \ln(1 - y/y_o) = (t/\gamma)^{\beta} \qquad 5.2$$

Consequently,

$$\ln \frac{1}{1 - y/y_o} = \frac{t}{\gamma}^{\beta} \qquad 5.3$$

If the logarithm of both sides of Equation 5.3 are taken, then

$$\log.\ln \frac{1}{1 - y/y_o} = \beta\log.\left(\frac{t}{\gamma}\right) \qquad 5.4$$

from which an expression for a straight line can be evaluated, viz,

$$\log.\ln \frac{1}{1 - y/y_o} = \beta(\log.t - \log.\gamma) \qquad 5.5$$

Re-arranging,

$$\log.\ln \frac{y_o}{y_o - y} = \beta\log.t - \beta\log.\gamma \qquad 5.6$$

The constants β and γ can be determined for each experimental run by carrying out a linear regression analysis using data for $\log.\ln \frac{y_o}{y_o - y}$ and log.t.

The constant β is related to the shape of the adsorption wave and γ corresponds to a time when 63% of the inlet concentration of the solvents is registered in the exhaust stream from the adsorber. The shorter this time, then the more vertical is the mass transfer zone. It therefore follows that the smaller the value for γ, the greater the mass transfer coefficient, and it can be shown that,

$$\frac{1}{\gamma} \; \alpha \; K_{OG}$$

where K_{OG} represents an overall mass transfer coefficient.

Data Tables of $\log.\ln \frac{y}{y_o - y}$ and log.t used in the linear regression analyses for determining the constants β and γ with regard to the different experimental operations are listed in Appendix IV.

5.3.1 Single Adsorption Operations

5.3.1.1 Perchloroethylene

A summary of the constants obtained for certain operations involving the adsorption of the chlorinated solvent vapour are listed in Table 5.2.

Run Number	Slope β	Intercept $\beta \log \gamma$	γ (seconds)	Deviation from Perfect Fit (Perfect Fit = 1)
BP1	1.8167	- 7.459	12758	0.9920
BP4	4.4730	-16.506	4900	0.9978
BP6	1.1059	- 3.373	1122	0.9106
BP10	2.4395	- 9.450	7477	0.9262
BP12	4.4687	-19.063	18445	0.9757
BP13	3.0448	-11.922	8232	0.9530
BP14	4.1896	-16.236	7504	0.8763

Table 5.2 - Values of β and γ for certain operations involving adsorption of perchloroethylene vapour

The application of Equation 5.1 as suggested by Doulah[95], for describing the adsorption wave, appears to be valid here since the deviations from the perfect fit straight line are small.

The data shown in Table 5.2 shows that γ decreases for both the increasing solvent concentration and increasing superficial gas velocity. This therefore infers that the mass transfer coefficient for the above system is proportional to both the perchloroethylene concentration and the velocity of the vapour stream. However, there appears to be no apparent correlation between the superficial gas velocity, perchloroethylene concentration and β.

5.3.1.2 Toluene

The constants β and γ evaluated for particular operations investigating the uptake of toluene vapour by charcoal cloth are outlined in Table 5.3.

Run Number	Slope β	Intercept β log γ	γ (seconds)	Deviation from Perfect Fit (Perfect Fit = 1)
BT1	3.2241	-13.866	19986	0.9745
BT5	2.0343	- 7.841	7151	0.9765
BT7	1.6348	- 5.904	4087	0.9554
BT8	3.7117	-15.831	18414	0.9127
BT9	0.9884	- 3.790	6831	0.9907
BT10	2.9281	-11.038	5864	0.9966

Table 5.3 - Values of β and γ for certain operations involving adsorption of toluene vapour

The deviations from the perfect fit straight line are again small here suggesting that by substituting the necessary constants listed in Table 5.3, Equation 5.1 can be used to describe the various adsorption waves.

As for perchloroethylene, the data infers that the mass transfer coefficient is proportional to both increasing superficial gas velocity and solvent concentration in the air. It is also shown in Table 5.3 that β decreases as the toluene concentration is reduced, but there appears to be no evident relationship between β and the velocity of the vapour.

5.3.2 Cyclic Adsorption Operations

The values determined for β and γ for the cyclic operations involving the adsorption of both perchloroethylene and toluene vapours are reported in Table 5.4.

With regard to perchloroethylene, a relatively high value of γ, 7158 seconds, has been computed for the first operation and a value of 4450 seconds was obtained for the second cycle followed by a gradual reduction in the values for γ in the third and fourth cycles. In comparison with the operations using toluene, the highest value of γ was again determined

Run Number	Slope β	Intercept $\beta \log \gamma$	γ (seconds)	Deviation from Perfect Fit (Perfect Fit = 1)
CBP1	2.4458	9.428	7158	0.9520
CBP2	1.7109	6.242	4450	0.9715
CBP3	1.5054	5.422	3995	0.9419
CBP4	1.5706	5.578	3560	0.9579
CBT1	1.6096	5.841	4254	0.9801
CBT2	1.9642	6.995	3641	0.9899
CBT3	1.5383	5.474	3618	0.9932
CBT4	2.0865	7.530	4064	0.9733
CBT5	1.6160	5.838	4098	0.9940

Table 5.4 - Value of β and γ for cyclic adsorption operations

for the first cycle, but then fluctuating values were obtained for an increasing number of cycles. It is also noted that with the exception of run CBP1, there is little difference between the values obtained for γ in all the other operations.

The highest value for β was evaluated for run CBP1, and for the subsequent cycles involving perchloroethylene, the β value fluctuates only slightly. Additionally, there is relatively little variation of β for all the cyclic operations involving toluene, thus suggesting adsorption waves of similar shape.

5.4 Rate of Mass Transfer

It is pointed out in Section 3.2.1.2.2 that Collins[79] put forward an expression for evaluating the length of unused bed at breakthrough, L, viz,

$$L = D - \left(\frac{G_s \, t_\beta \, (y_o - y_o^*)}{\rho_s \, (W_e - W_o)} \right) \qquad 3.17$$

The length of unused bed determined at the breakthrough point can be related to the total resistance to mass transfer. It follows, therefore, that the greater the length of unused bed at breakpoint, the less effective is the adsorptive system. The expression suggested by Collins has been applied to calculate L for selected operations undertaken on the batch system and the results are outlined below. Tables of data substituted into Equation 3.17 for the particular operations are included in Appendix V.

5.4.1 Single Adsorption Operations

Values of L as determined by Equation 3.17 and the fraction of unused adsorbent bed length at breakthrough for the selected single adsorption operations are reported in Table 5.5.

Run Number	Length of Unused Bed at Breakpoint, L - (m)	Fraction of Unused Bed Length at Breakpoint
BP1	0.4178	0.696
BP4	0.3226	0.538
BP6	0.4864	0.811
BP10	0.3242	0.539
BP12	0.1715	0.286
BP13	0.4008	0.668
BP14	0.2839	0.473
BT1	0.1409	0.235
BT5	0.0810	0.135
BT7	0.5172	0.862
BT8	0.0128	0.021
BT9	0.5364	0.894
BT10	0.3396	0.566

Table 5.5 - Values of L and fraction of unused bed at breakthrough for selected single adsorption operations

Since it is regarded that the smaller the length of unused bed, the more effective is the adsorptive system, then the most favourable conditions occur at the lowest superficial gas velocity. This is represented by BP12 for perchloroethylene and BT8 in the case of toluene.

It is evident from this data that the fraction of unused adsorbent bed at breakpoint differs significantly with experimental conditions. There also appears to be no apparent correlation between the length of unused bed (as determined by Equation 3.17) and both the solvent concentration and the superficial gas velocity. It is assumed that this is partly due to the time to breakthrough being crudely estimated from the breakthrough curve. Additionally, the application of Equation 3.17 may not be valid here.

5.4.2 Cyclic Adsorption Operations

Values of L as determined by Equation 3.17 and the fraction of adsorbent bed length unsaturated at breakthrough with respect to the cyclic adsorption operations are shown in Table 5.6.

Run Number	Length of Unused Bed at Breakpoint, L - (m)	Fraction of Unused Bed Length at Breakpoint
CBP1	0.0720	0.120
CBP2	0.4032	0.672
CBP3	0.3890	0.648
CBP4	0.4238	0.706
CBT1	0.2845	0.474
CBT2	0.1527	0.254
CBT3	0.1844	0.307
CBT4	-0.2208	-

Table 5.6 - Values of L and fraction of unused bed length at breakthrough for cyclic adsorption operations

The data used to evaluate L is summarized in Table 2, Appendix V. With the exception of run CBT4, Y_o^* is assumed to be zero since such values are negligible for the corresponding values of W_o. The apparent density takes into account the mass of solvent adsorbed at the start of each operation.

A relatively small fraction of unused bed length was obtained at the breakthrough point for the first operation involving the adsorption of perchloroethylene. This increased to between about 65% and 70% for the subsequent runs. However, the converse is apparent with regard to the cyclic adsorption of toluene. This further evidence also suggests that the equation put forward by Collins[79] used to express the rate of mass transfer in terms of the length of unused bed cannot be applied to the adsorptive systems described above.

CHAPTER 6

CONTINUOUS RIG OPERATIONS AND RESULTS

PLATE 6.1

GENERAL VIEW OF EXPERIMENTAL EQUIPMENT USED FOR THE CONTINUOUS OPERATIONS

1	Pressurized Air Supply Line
2	Syringe Pumps
3	Electric Fan
4	Adsorber
5	Charcoal Cloth
6	Water Reservoir
7	Boiler
8	Superheater
9	Regenerator
10	Mass Spectrometer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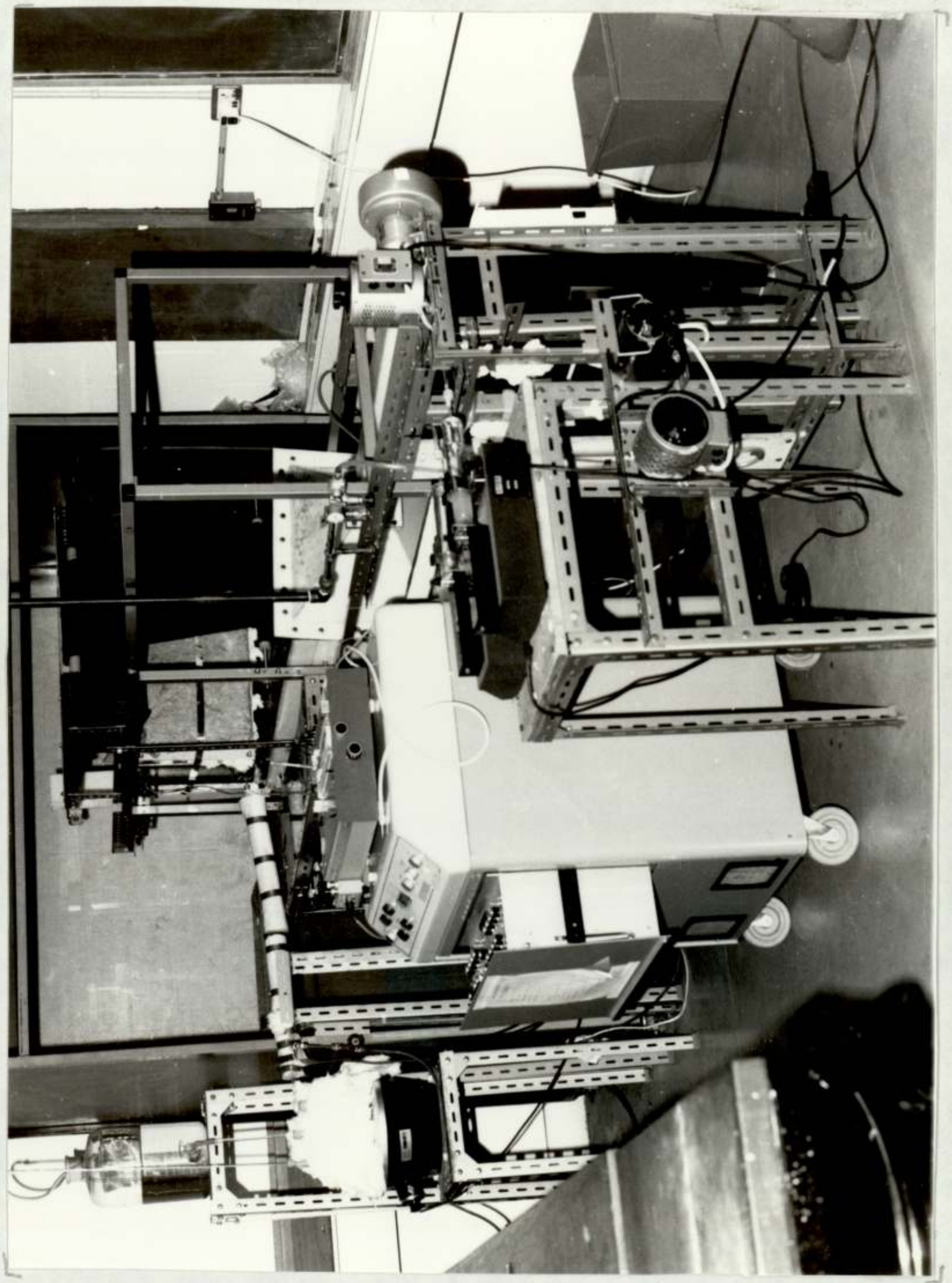
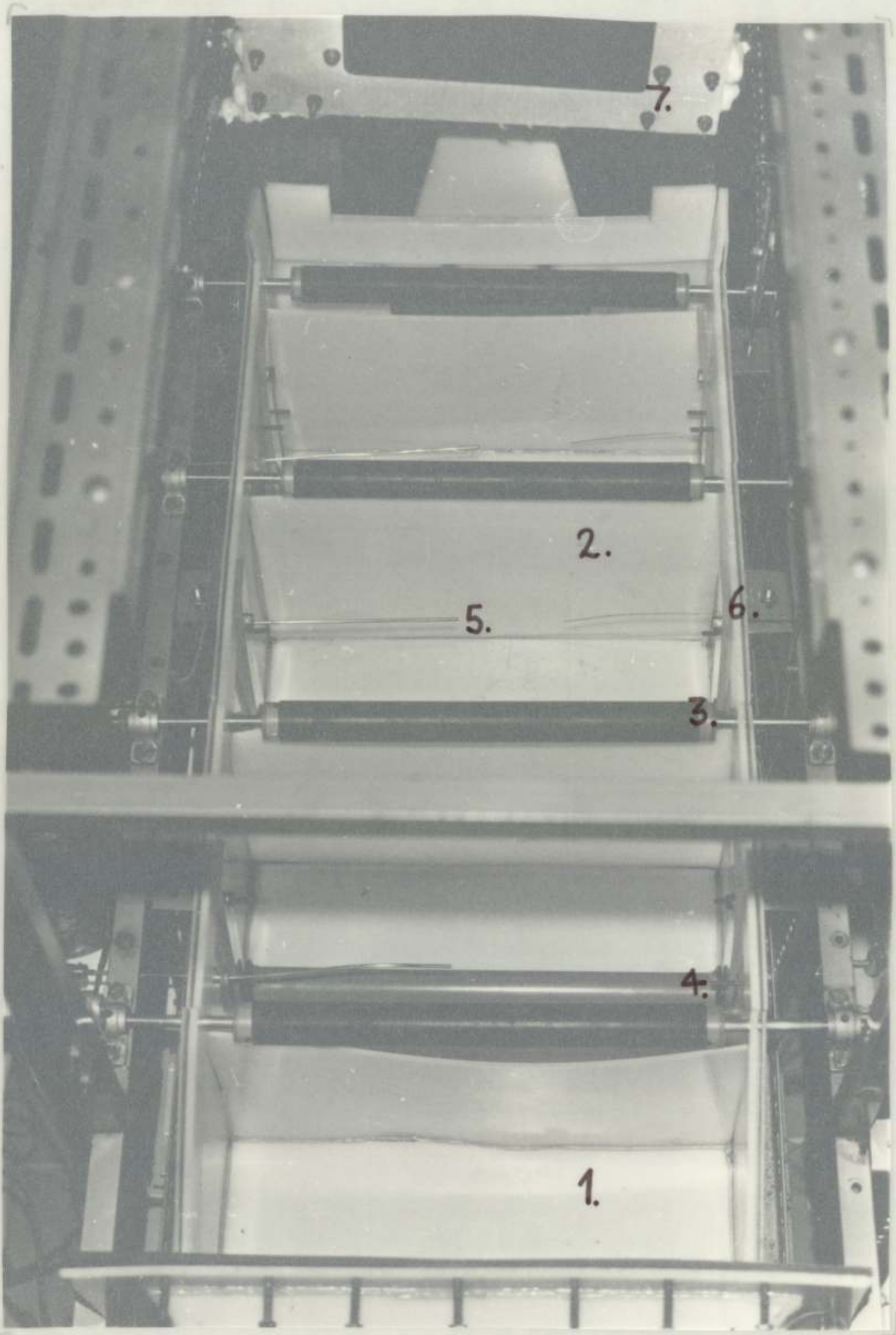


PLATE 6.2

ARRANGEMENT OF ROLLERS IN THE ADSORBER

1	Adsorber
2	Baffle
3	Neoprere-Rubber Coated Driven Roller
4	Undriven Roller
5	Thermocouple
6	Sample Point Probe
7	Regenerator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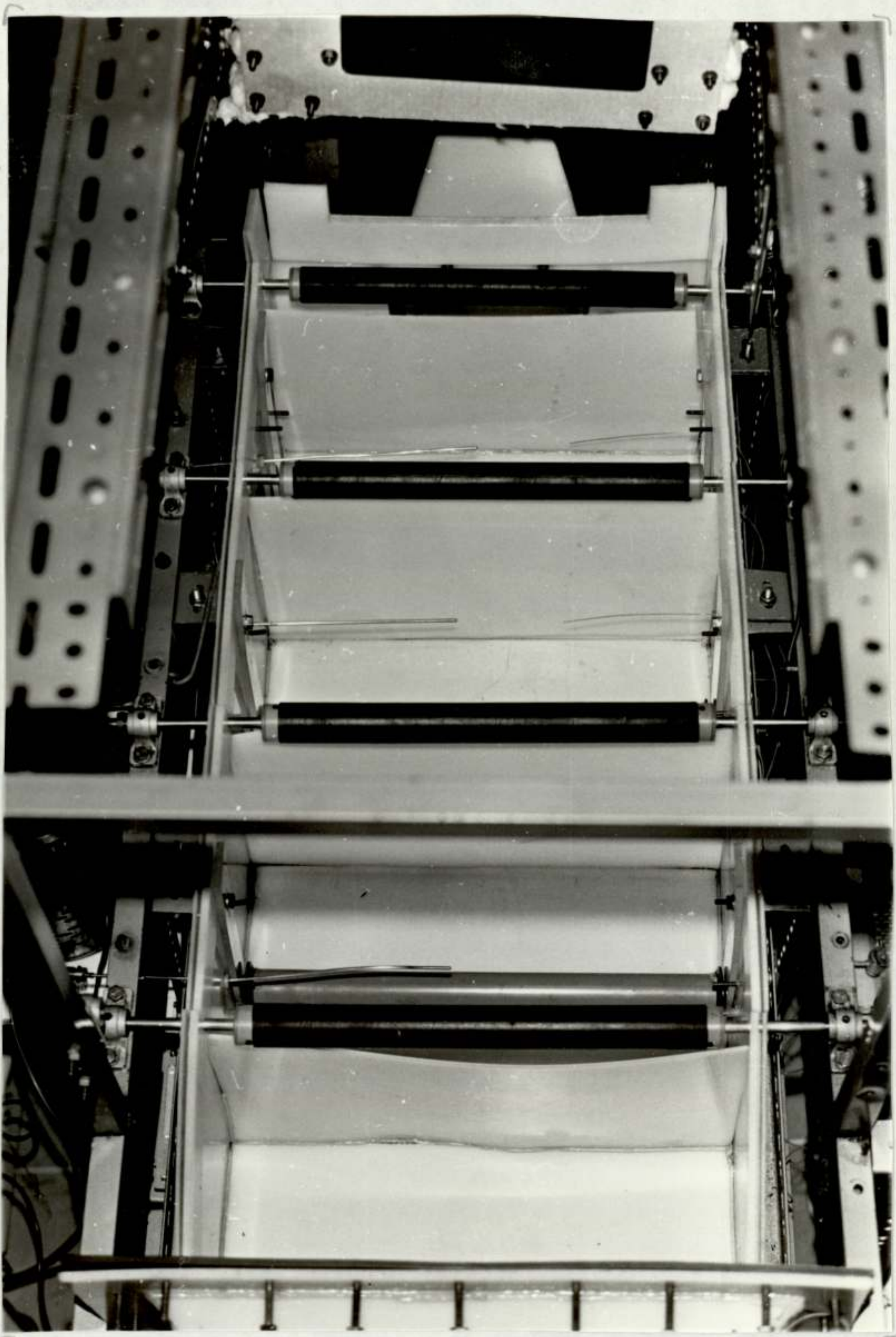

6 CONTINUOUS RIG OPERATIONS AND RESULTS

Although activated charcoal cloth does not appear favourable with regard to the batchwise recovery of organic solvent vapours, it was believed that charcoal cloth could be exploited in a continuous system. The type of set-up intended to achieve this is outlined in Section 6.1. A description of the operations undertaken on the continuous rig and a discussion of the results are then highlighted. The effect of the number of regeneration cycles on the charcoal cloth's adsorbent life has been investigated by Smith[100] at CDE Porton Down. Details of the tests carried out and their results are included in Section 6.3. This Chapter finally concludes with recommendations for improvements to the continuous rig and puts forward suggestions for alternative designs for the continuous recovery of organic vapours.

6.1 Description of Continuous Rig

6.1.1 General Description of Process

The main features of the continuous system shown in Plate 6.1 and represented diagrammatically in Figure 6.1 are now described.

The continuous set-up basically consists of four sections: an air-solvent mixing section, an adsorbing region, a regenerator and a solvent recovery unit.

Liquid solvent is vapourized over a 'hot finger' (as for the batch system) before being carried by an air stream to the air duct where it is mixed with a much larger volume of flowing air which is drawn from the atmosphere by means of an electric fan. The air solvent mixing section is depicted in Figure 6.2.

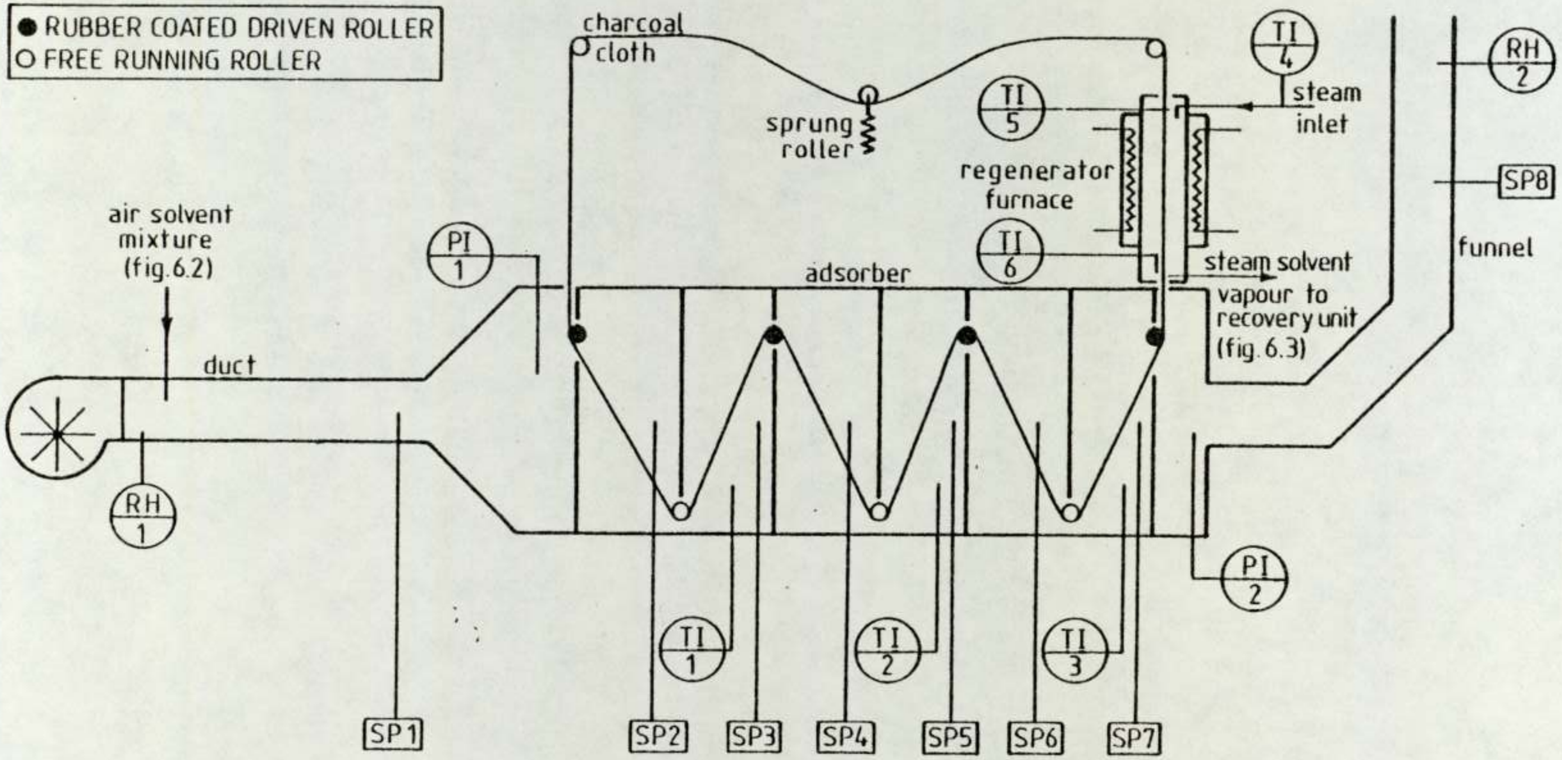

FIGURE 6.1 - SCHEMATIC DIAGRAM OF EXPERIMENTAL APPARATUS USED FOR THE CONTINUOUS OPERATIONS

The solvent-laden air stream then passes to the adsorbing region where the vapour stream is brought into contact with the charcoal cloth. The cloth can be driven around the rollers either counter-currently or co-currently. The arrangement of the rollers is shown in Plate 6.2. The exhaust from the adsorber is discharged through a funnel and extracted from the laboratory via a fume hood.

The solvent-laden cloth passes into the regenerator at its base if the continuous system is being operated co-currently or, if counter-currently, through the top. Two electric heaters fitted to the sides of the vessel are available for pre-heating and minimizing any condensation inside the vessel. Steam used for stripping the solvent from the cloth is generated in situ and then passed to the regenerator.

The steam-solvent vapour mixture extracted from the regenerator by a vacuum pump is condensed before being collected in a flask. A diagram of the solvent recovery system is included in Figure 6.3.

If the cloth is being driven co-currently, it passes through the top of the desorber and, as it moves through the atmosphere, it is cooled and dried before being returned to the first section of the adsorber. Alternatively, if the charcoal cloth is moving counter-currently, it leaves the regenerator at its base and immediately passes into the final section of the adsorption unit to continue the process.

The four major units constituting the continuous rig are now described in more detail.

6.1.1.1 Solvent-Air Mixing Section

In order to maintain a continuous operation, the solvent vapour-air mixing unit depicted in Figure 6.2 has two syringe pumps and two glass hot fingers, and only one syringe pump and glass finger are in use at any

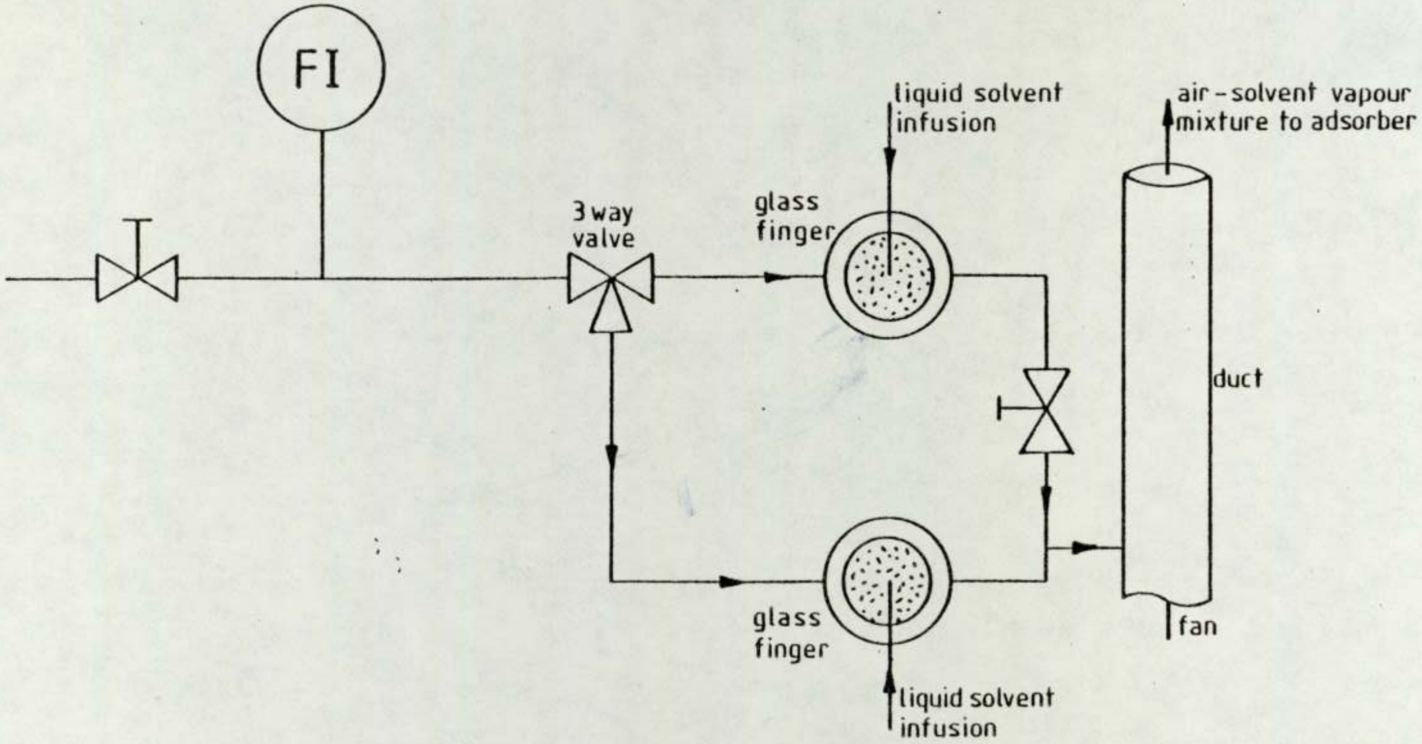

FIGURE 6.2 - AIR - SOLVENT MIXING SECTION

one time. The syringe pumps used to deliver the liquid solvent and the hot finger on which it is vapourized are similar to those described in Section 4.2.1.1. Air flowing at 1.72×10^{-4} $m^3.s^{-1}$ from a pipe line in the laboratory carries the organic vapour to the duct where it mixes with the bulk of the air.

The bulk air stream can be varied between rates of 0.0063 $m^3.s^{-1}$ and 0.0231 $m^3.s^{-1}$ by adjusting the electrical input to the fan. A wide range of solvent vapour concentrations can be manufactured by changing the rate at which the liquid solvent is vapourized and the flow of air discharged by the fan. The volumetric concentration of perchloroethylene can be set at values between 0.0025% and 0.0922%, and in the case of toluene, from 0.0024% up to 0.0862%.

The solvent vapour and the air continue to mix as they pass 1.0m down a high density polyethylene (HDPE) rectangular duct (measuring 0.104m by 0.084m) before entering the adsorber section.

6.1.1.2 <u>Adsorber Section</u>

The adsorber, also made from HDPE, is essentially a duct divided into six sections by baffles. The duct itself is rectangular measuring 0.424m by 0.348m and is 1.000m long. The baffles are fitted to enhance turbulence and direct the vapour stream such that it flows parallel to the charcoal cloth.

It is shown in Figure 6.1 that four rollers are positioned above the lower baffles. Each of these rollers is individually driven to give a constant moving rate through the whole continuous system. The rollers are rubber coated to prevent any slippage and each one is mounted on a 0.0095m diameter shaft. The shafts are driven by a chain mechanism by a 5 rpm motor. The remaining rollers are free running.

The charcoal cloth fitted around the rollers was generally 0.30m wide and 3.43m long. The ends of the cloth which overlapped by about 0.10m were satisfactorily attached by 'Evostick' adhesive compound. The speed with which the cloth passes through the adsorber could be varied between speeds of 0.0023 and 0.00248 $m.s^{-1}$ by a variable speed control system.

6.1.1.3 Regenerator and Solvent Recovery Systems

The regenerator included in Figure 6.3 consists of a rectangular stainless steel enclosure of dimensions 0.425m x 0.335m x 0.132m. Steam, available at rates between 1.60×10^{-5} and 1.85×10^{-4} $kg.s^{-1}$ is introduced through an upper point on one of the smaller sides of the regenerator. The steam is generated in situ, the boiler and superheater being similar to those described for the batch rig in Section 4.2.1.3. As stated previously, in order to pre-heat the desorber and minimize condensation, two 2.3kw heaters are fitted to the two larger sides of the vessel. The regenerator is insulated by both kaowool and 'Sindanyo' slabs. However, to deal with any condensation, the base of the regenerator is slightly inclined and any liquid can be drained from the vessel via two valves. The regenerator has two narrow slots, one at its base and the other at the top so that the charcoal cloth can pass through. The slots are fitted with rubber flaps in an effort to seal the system.

A vacuum pump is employed to withdraw the steam-solvent vapour mixture from the regenerator. The outlet is situated near the base of the vessel, diagonally opposite the steam inlet. In doing so, the steam is encouraged to pass through the cloth as well as bringing it into contact with as much charcoal cloth area as possible.

The extracted vapour mixture is condensed and cooled by cold water before finally being collected in a flask. A cold finger trap, as depicted in

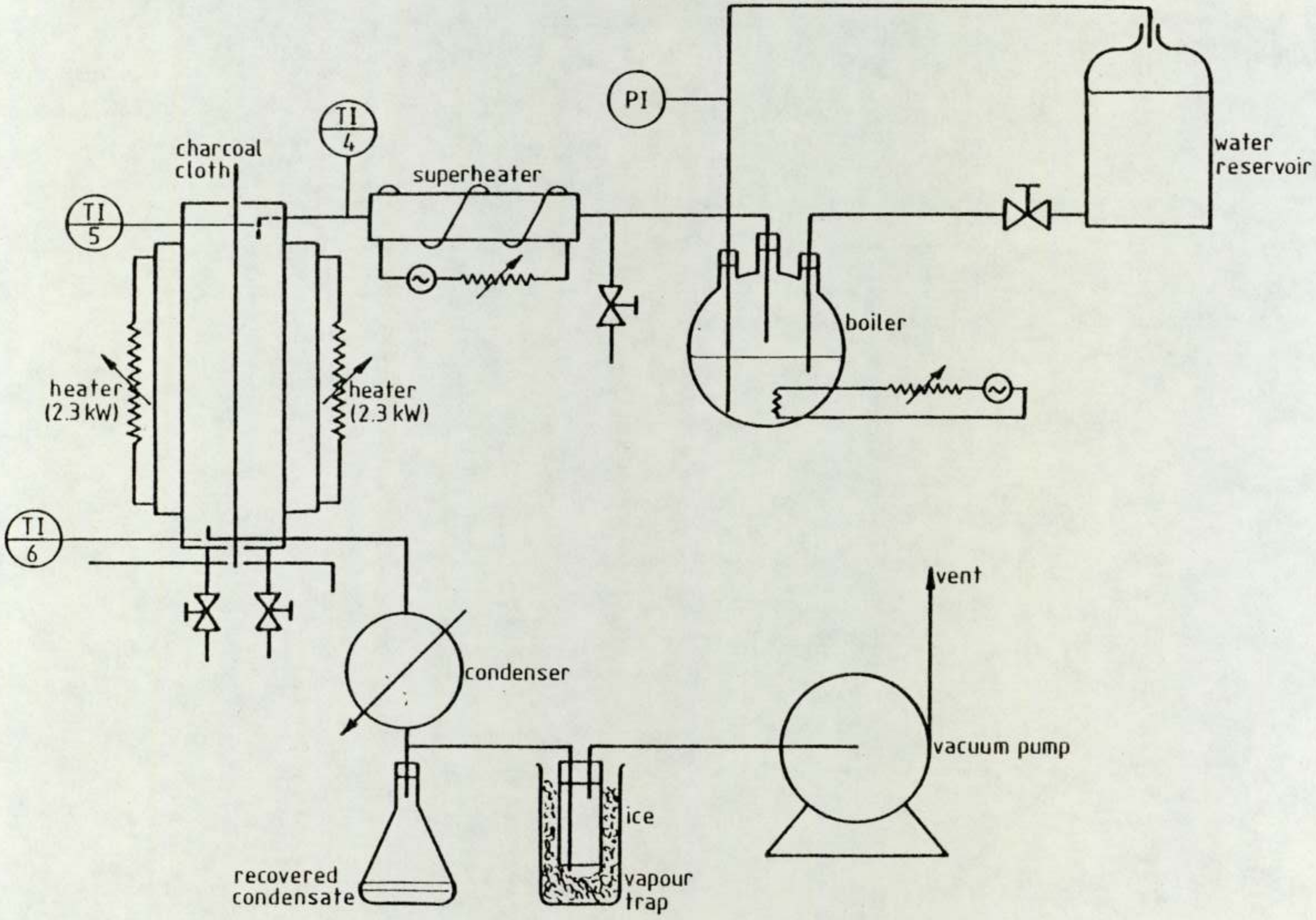

FIGURE 6.3 - REGENERATOR AND SOLVENT RECOVERY SYSTEMS

Figure 6.3, is fitted between the collection flask and the vacuum pump to prevent any uncondensed vapour being vented.

6.1.2 Instrumentation and Analysis

The instrumentation associated with the continuous system and the location of the sample points are shown in Figure 6.1.

The relative humidity was determined from wet and dry bulb thermometer readings; the humidity of the inlet air was taken to be that of the atmosphere and a wet and dry bulb thermometer was placed in the funnel so that the outlet humidity could be registered. The pressure drop across the adsorber section was measured using water manometers; one limb of each manometer being open to the atmosphere and the other being connected in the adsorber. The temperature at six points in the continuous system was measured using thermocouples. Three thermocouples were used to register the temperature at the front, middle and end sections of the adsorber, the temperature of the steam fed to the desorber was also recorded and two thermocouples were attached to the regenerator so that the inlet steam and the vapour mixture leaving the vessel could be monitored.

The concentration of the solvent vapour was again measured using a mass spectrometer. Specimens of the vapour mixture were withdrawn via a syringe needle and plastic capillary tube and the analysis details of the read-out were similar to those described in Section 4.2.2.1 for the batch rig. The mass spectrometer probe was placed at the edges of the adsorber as well as at its centre for sample points 2 - 7 inclusive, so that any concentration variations along the width of the duct could be studied.

During each operation condensate was collected regularly. Since the solvents, perchloroethylene and toluene, are immiscible with water, the quantity of each liquid could simply be monitored by pouring the condensate into a measuring cylinder.

6.2 Experimental Conditions and Results

The experimental conditions and results are discussed separately for each operating run. This is because the results from the first run, designated CP1, decided the objectives for the subsequent operation, CP2A, the results of which influenced the conditions for run, CP2B, and so on. A summary of the results from these operations is tabulated in Appendix VI.

6.2.1 Perchloroethylene

6.2.1.1 Run CP1

The objectives of this run were essentially to test the system and gain some indication of the effectiveness of perchloroethylene recovery.

A low air rate of 6.47×10^{-3} $m^3.s^{-1}$ and a solvent vapour rate of 3.18×10^{-6} $m^3.s^{-1}$ were used; the volumetric concentration of the perchloroethylene vapour in the air being 0.0492%. The average cloth speed was 2.38×10^{-3} $m.s^{-1}$ and since a low air rate was used, the contact time between the solvent vapour and the charcoal cloth was relatively high. The temperature inside the adsorbing section was 25 ± 2^0C, the temperatures being slightly higher near the outlet. By adjusting the electrical heat input to the superheater and regenerator, the temperature inside the regenerator was maintained between 110^0C and 130^0C in an attempt to prevent any condensation inside the vessel.

The adsorption results are shown in Table 1, Appendix VI, and all the concentrations recorded corresponded to the probe being placed at the edge of the duct. At best, 71.0% of the perchloroethylene vapour was adsorbed and this compares with complete adsorption of the solvent by the batch system. The outlet solvent vapour concentration fluctuated with a gradual increasing tendency with adsorption time. The data summarized in the Table also shows that the solvent concentration decreases along the length of the adsorber duct, and the concentration at a particular sample point also appears to increase with adsorption time, although the data on this are limited. Since the mass spectrometer used to analyse the gaseous mixture has only one probe and it takes almost one minute to measure the solvent concentration at any one position, it is therefore difficult to obtain a representative concentration profile at any instant.

No solvent at all was recovered and only 1.6. x 10^{-5} m^3 (16.10ml) of water was condensed during 7200 seconds (2 hours).

6.2.1.2 Runs CP2

Run CP2 has been divided into three separate experiments, the different sets of operations being undertaken on the same piece of charcoal cloth. The adsorption results from these operations have been reported in Table 2.

6.2.1.2.1 Run CP2A

The objective of this experiment was to test the reproducibility of the results from CP1. The perchloroethylene concentration in the air was again set at 0.0492%, the air and solvent vapour rates being unchanged and the cloth speed was 2.392 x 10^{-3} $m.s^{-1}$. The temperature inside the adsorber was initially uniform at 22^0C, but increased to 27^0C and 30^0C at the inlet and outlet respectively, this being due to some heat transfer from the regenerator.

The adsorber process was continued for about one hour, and during this time approximately half the solvent vapour was adsorbed constantly. The longitudinal concentration profile again shows a gradual fall in perchloroethylene vapour concentration towards the adsorber outlet. None of the chlorinated solvent was recovered. Water was collected at a rate of 4.90 x 10^{-9} $m^3.s^{-1}$ and since steam was fed into the regenerator at a rate of 1.60×10^{-8} $m^3.s^{-1}$, it appears that the majority of the steam remained adsorbed on the cloth.

6.2.1.2.2 Run CP2B

The cloth used in this investigation was the same as that used in Run CP2A, but had been left on the rig overnight. The objective was to determine the time it took for the charcoal cloth to become saturated with perchloroethylene solvent without any regeneration. (This experiment therefore does not constitute continuous solvent recovery.)

The highest volumetric perchloroethylene concentration possible at 0.0615% was used, the lowest air rate and by setting the charcoal cloth at a speed of 2.29×10^{-3} $m.s^{-1}$ meant that the contact time between the charcoal cloth and solvent vapour was at a maximum. The relative humidity of the inlet air was 68% and the temperature variations inside the adsorber were similar to those outlined for Run CP2A.

The cloth took some 9480 seconds to become saturated under these conditions, and after this time the concentration of the solvent in the exhaust was greater than that at the inlet. It therefore appears that some of the solvent vapour was desorbed from the cloth by the vapour stream. The adsorption results in Table 2 once more show a gradual decrease in perchloroethylene vapour concentration from the inlet to the outlet of the adsorbing section.

6.2.1.2.3 Run CP2C

Once the above cloth was saturated, it was regenerated by infusing steam at a rate of 8.61×10^{-8} $m^3.s^{-1}$ for 1380 seconds before the solvent was injected once more (hence resuming to continuous operation).

The results in Table 2 show that initially some 23.9% of the inlet perchloroethylene vapour had to be removed by the time the vapour stream had reached the inlet, and this improved slightly with increased adsorption time. Although solvent had clearly been desorbed from the cloth, none was recovered. It is thought that the rate of ingress air withdrawn from the regenerator by the vacuum pump is such that the concentration of the solvent vapour is so small that the vapour pressure at the condensation temperatures remains well below its saturation vapour pressure.

6.2.2 Toluene

Although evidence from tests undertaken using perchloroethylene solvent clearly suggests that the continuous system requires some major modifications if it is to recover solvent at efficiencies compared with those obtained during the batch operations, experiments were carried out to study the adsorption of toluene vapour. These were done so that the concentration profiles could be studied against those obtained for the chlorinated solvent. A further objective was to compare solvent concentrations at the centre as well as at the edge of the duct.

6.2.2.1 Runs CT1

Two experimental runs CT1A and CT1B were carried out using the same section of charcoal cloth, the adsorption results being summarized in Tables 3 and 4 respectively. It is noted here that the symbol E refers to the probe being positioned at the edge of the duct and M, in the centre. The objectives, experimental conditions and the results obtained with respect to these two operations are now described.

6.2.2.1.1 Run CT1A

The volumetric concentration of the toluene vapour was maintained at 0.0231% and the speed of the charcoal cloth averaged 2.29×10^{-3} m.s^{-1}.

The adsorption temperature was 23 ± 1^0C, the relative humidity of the inlet air was constant at 66% and the effluent varied from initially 58% up to 75%. The external heaters attached to the desorber were not switched on in an attempt to encourage the vapours to condense inside the regenerator itself. The temperature inside the regenerator was 59 ± 2^0C and the steam was introduced at 108^0C.

The results in Table 3 show that the outlet toluene concentration fluctuated between 0.0116% and 0.0179%, the corresponding fractions of the inlet concentration being 0.503 and 0.777. The solvent concentration is also seen to be higher at the edges of the duct than in the middle. In one case as measured at sample point 4, the amount of toluene at the sides of the duct is more than twice that in the centre, yet in other locations such as at sample point 5, there is only slightly more solvent at the edges. Although the data are limited, it does appear that as in the operations involving perchloroethylene, the concentration of the solvent vapour in the air gradually decreases towards the adsorber outlet. However, the last batch of results involving samples being taken from positions 2, 5 and the outlet show that there is little concentration variation throughout the adsorber.

The cloth was regenerated for a total of 8700 seconds and during this time, the condensate, consisting only of water, was recovered from both the collecting flask and the desorber itself. Some 2.4×10^{-5} m^3 of water was recovered in the flask and a total of 10.41×10^{-5} m^3 was withdrawn from the regenerator. In contrast with desorption being undertaken with the external heaters being switched on, an appreciable higher amount of condensate was recovered. However, it still remained significantly below the volume of steam fed to the vessel, thus suggesting steam losses as well as quantities being taken up by the charcoal cloth.

6.2.2.1.2 Run CT1B

The main objective was to attempt for a steady state condition in that the concentration of the solvent in the exhaust remained constant.

The cloth used in this test was the same as that in CT1A. The air flow rate was 6.47×10^{-3} $m^3.s^{-1}$ and the toluene vapour concentration in the air was again 0.0231%. The mean adsorption temperature was 21.5°C. The relative humidity of the air at the inlet was 63% and at the outlet, 58%. The latter increased to 75% once the steam had been introduced for desorption. This therefore suggests that the charcoal cloth was taking up water vapour from the air, but during regeneration the readings imply that the air was stripping water vapour from the cloth.

The adsorption results summarized in Table 4 show that the toluene concentration within the adsorber fluctuates with time in relation to the longitudinal position as well across the width. The concentration of the organic solvent in the effluent changed between 0.0139 and 0.0162% represending 60 and 70% of the inlet volume respectively, during approximately the initial 1.8×10^4 seconds. However, after this time, the concentration had increased to 0.0199% which was higher than the volumetric concentrations upstream of the funnel. With regard to sample point 4, the solvent concentration in the middle of the duct was less than at the sides, but at sample points 2 and 3, the contrary was recorded. There is insufficient evidence to draw any conclusion from these concentration variations, and consequently more data are necessary before any trends can be established.

The heat for regeneration was again provided solely by the steam. Although an equivalent total of 2.17×10^{-3} m^3 of water had been fed to the desorber, only 1.46×10^{-4} m^3 was recovered from the regenerator and 0.815×10^{-4} m^3 from the collecting flask. The desorption process was carried out for 3.12×10^4 seconds. It is therefore evident that relatively large quantities of steam are being lost and adsorbed by the charcoal cloth.

6.3 Examination of the Effect of the Number of Regenerations upon the Activity of Activated Charcoal Cloth

6.3.1 Introduction

The residual activity of charcoal cloth samples used in the recovery of the vapour phase solvents was investigated by Smith and Davies[100] at CDE Porton Down. This was achieved by determining the heats of wetting of the charcoal cloth as well as measuring the saturation vapour uptake. The sample treatment is listed in Table 6.1.

Sample Number	Number of Regenerations
1, 2, 3	Controls
4, 5, 6	1
7, 8, 9	7
10, 11, 12	15
13, 14, 15	21

Table 6.1 - Sample Numbers and corresponding regenerations used in determining residual activity of charcoal cloth

6.3.2 Experimental Methods

The heat of wetting tests were carried out in the Porton Heat of Wetting Calorimeter[100], employing benzene and silicone as the wetting liquids.

Dried, weighed samples of charcoal cloth were exposed to the saturated atmospheres of two test vapours, benzene and carbon tetrachloride in a dessicator.

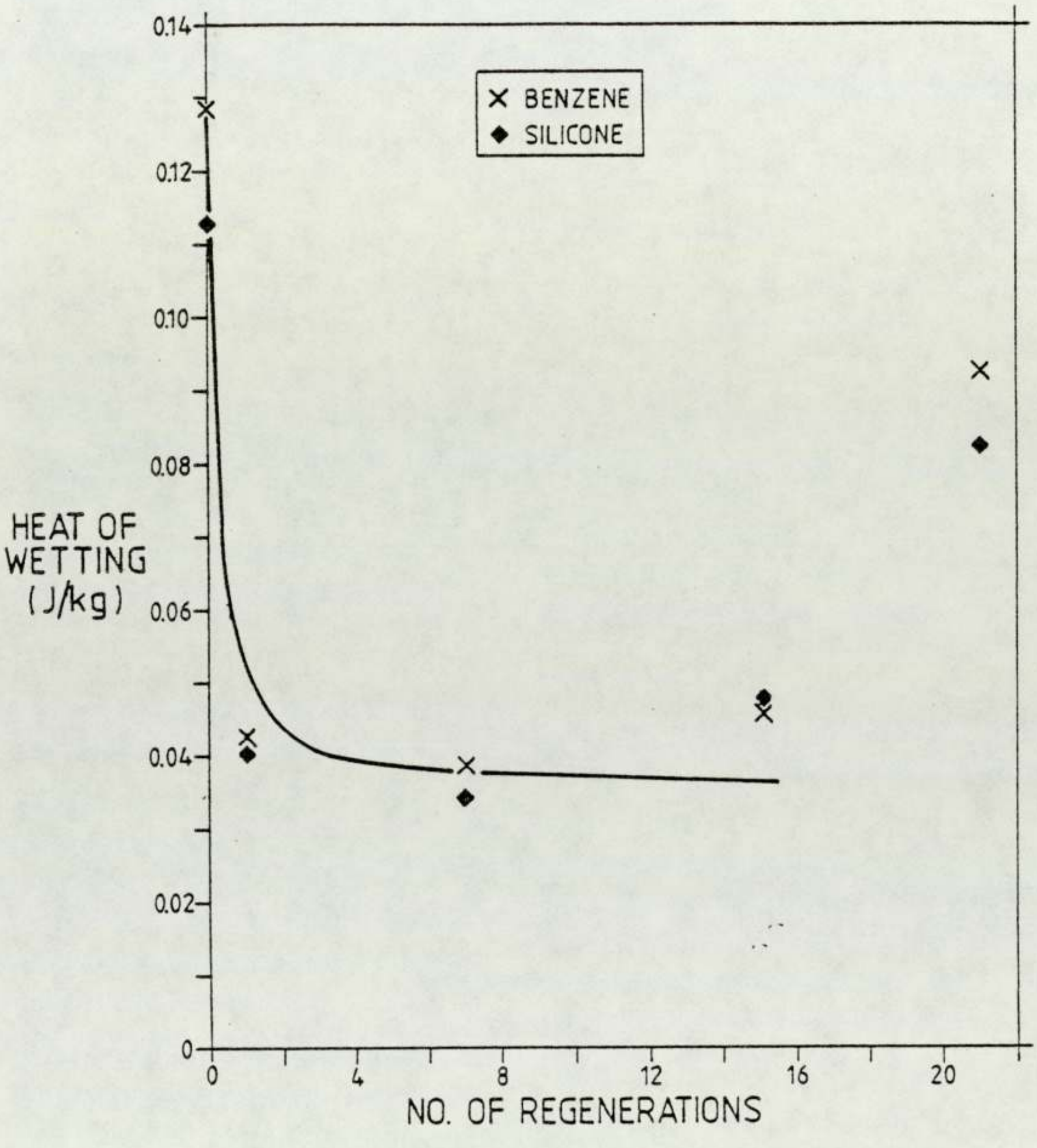

FIGURE 6.4 - VARIATION IN CHARCOAL CLOTH ACTIVITY WITH NUMBER OF REGENERATIONS

6.3.3 Results

The results from the heat of wetting and the saturation vapour uptake tests are reported in Table 1, Appendix VII. Samples 4 - 12 inclusive, involved regeneration after adsorption with perchloroethylene vapour and the results in Figure 6.4 show that after an initial drop in the heat of wetting after one regeneration, it then remains fairly constant with an increasing number of regenerations.

The charcoal cloth samples 13, 14 and 15 which had undergone 21 regenerations had been involved in the continuous recovery of toluene. The heat of wetting results are higher than for the previous specimens, inferring that the reduction in the residual activity of charcoal cloth is not as great when taking up toluene vapour. The results also indicate that at least 30% of the charcoal cloth activity remains after 15 regenerations when used for the adsorption of perchloroethylene vapours.

6.4 General Discussion

The results obtained from the operations undertaken on the continuous system in no way lend themselves to any theory or empirical relationships, but do serve as evidence which highlights the design problems associated with the continuous arrangement.

The effectiveness of adsorption is seen to be appreciably less than those obtained from the batch rig. In fact, complete adsorption was not achieved at any time. One possible reason for this could be that some of the air-solvent vapour mixture was by-passing the charcoal cloth. The solvent vapour concentrations taken particularly during the uptake of perchloroethylene indicate that the solvent concentration was less at the centre of the duct than at its edges. This therefore suggests that the adsorber duct should be fitted with deflector baffles such that vapour stream is directed towards the middle of the charcoal cloth. In order to prevent any egress vapour mixture being treated, it is recommended that the system be operated under a slight vacuum. This could be attained by 'sucking in' atmospheric air by fitting a pump to the adsorber outlet. It is also suggested that the length of the adsorber duct be increased in an attempt to improve the effectiveness of solvent vapour uptake.

Several minutes had to elapse before any representative measurement at any one point could be established; the number of solvent vapour concentration measurements were thus severely limited. Consequently, more elaborate analytical equipment is necessary before any trends can be established.

Although the tests showed that solvent was being desorbed from the cloth, none was actually recovered. This is believed to be a result of ingress air in the regenerator which is such that the partial pressure of the solvent vapour in this air did not exceed its vapour pressure under condensation conditions. Therefore, the regenerator needs to be completely sealed so that condensibles alone are withdrawn from the vessel.

It is intended that the suggested modifications to the continuous arrangement described above will improve its performance. However, the task of removing and replacing the cloth remains laborious. A proposed alternative is the set-up shown, in a basic schematic form, in Figure 6.5. The charcoal cloth fitted on a series of reels moves perpendicular to the solvent laden air stream. The reels are sealed in a vessel into which steam for regeneration, then air for drying the cloth are introduced. Replacing the cloth is simpler using this type of set-up and binding the cloth is unnecessary.

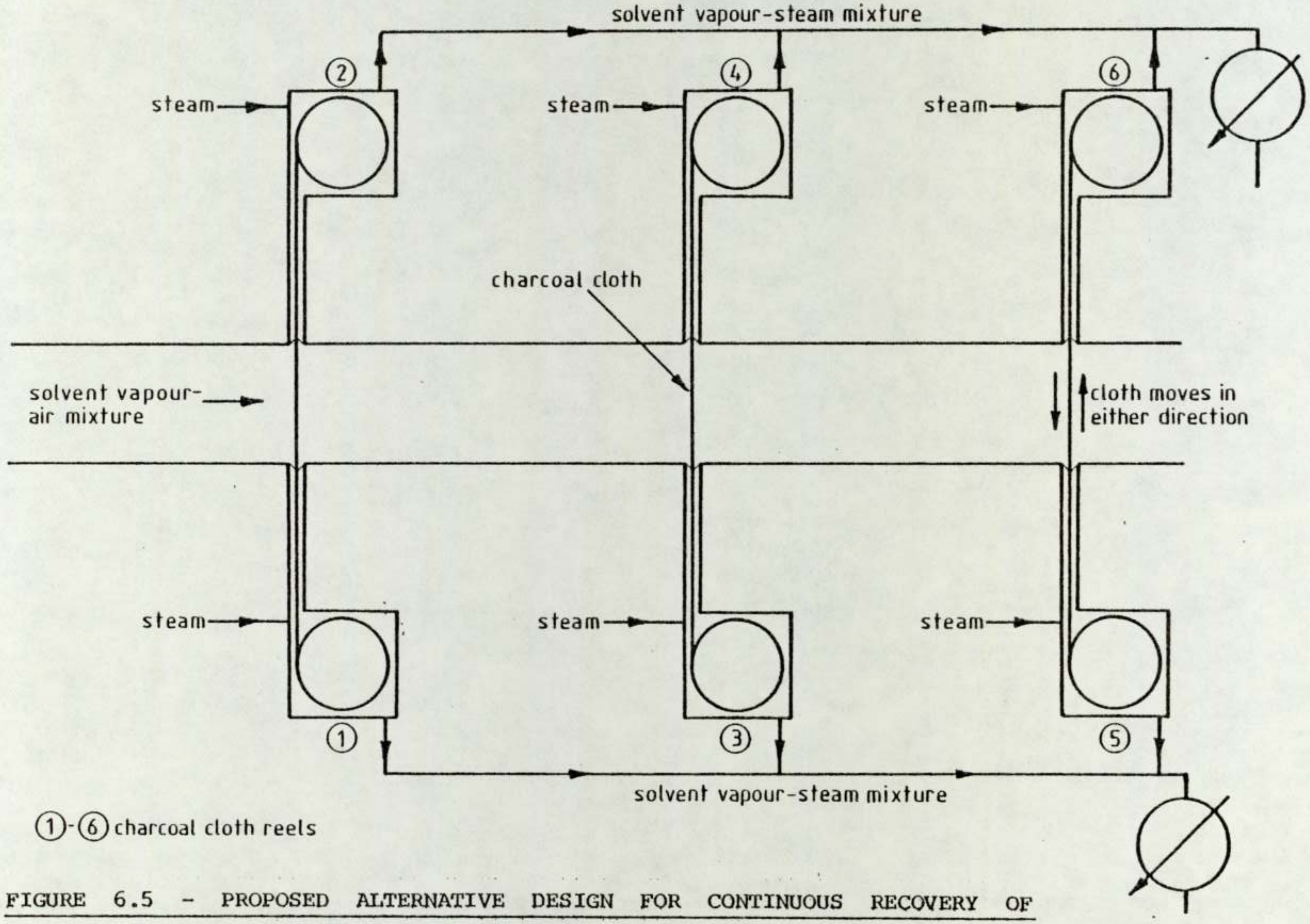

FIGURE 6.5 - PROPOSED ALTERNATIVE DESIGN FOR CONTINUOUS RECOVERY OF VAPOUR PHASE SOLVENTS USING ACTIVATED CHARCOAL CLOTH

CHAPTER 7

MARKET POTENTIAL FOR CHARCOAL CLOTH

7 MARKET POTENTIAL FOR CHARCOAL CLOTH

7.1 Introduction

Evidence in previous Chapters exists to demonstrate that activated charcoal in the form of charcoal cloth would not be favourable for the batchwise recovery of solvents. With regard to continuous recovery, further development work has yet to be undertaken before charcoal cloth can be established as an effective and economic material. Therefore, at present, granular charcoal appears to be the more suitable form of activated carbon for reclaiming organic solvents from vapour streams.

Activated charcoals in the granular and powdered forms have many uses and these are outlined. It is considered that activated charcoal cloth can be beneficial in a number of these fields and it is believed that the cloth also has potential in new market areas. The possible uses of charcoal cloth are discussed below under medical, defence, industrial and domestic applications and a separate Section also highlights the opportune usefulness of the material to combat static electricity.

7.2 Present Day Uses for Activated Charcoals

The demand for activated charcoal in the United States in 1976 was estimated at 8.35×10^7kg. The Table in Appendix VIII shows how the demand for activated carbon was distributed among the various markets and includes the projected average annual growth rates.

It is seen that liquid phase applications accounted for 85% of the total demand, the largest market being the sugar industry. However, it was anticipated that the highest growth areas for active charcoals would be for the treatment of waste water and drinking water.

The Table also shows that only 1½% of the total demand for activated carbons was used for the recovery of solvent vapours. Therefore, although at present charcoal cloth may not be viable for the recovery of valuable vapour phase solvents, there are still a large number of possible market areas where charcoal cloth could replace present forms of carbon. One particular usefulness is anticipated in the nuclear power industry to protect personnel and the environment from radio-active emissions. The physical state of activated charcoal cloth enables it to be conveniently fitted around the reactor core and it is believed that this will lead to simpler constructions than are used or considered at the present time.

Additionally, there may be market areas where the cloth could be used to replace other adsorbents such as alumina and zeolites.

It is important to note in studying industrial markets with regard to charcoal cloth that it is not merely concerned with selling or buying a new product, but a new process. Although powdered and granular carbons and activated charcoal cloth are all adsorbents, their physical natures differ considerably - consequently, their handling procedures will not be the same when used in a process. Charcoal cloth enables transport and handling without the losses of adsorbent due to abrasion and entrainment experienced by the granular and powdered varieties.

7.3 Market Areas for Charcoal Cloth

7.3.1 Medical

The flexible property of charcoal cloth means that it can be used with bandages as an aid to healing as well as adsorbing any offensive odours emanating from wounds. Operating personnel can also benefit from the flexibility of charcoal cloth when used in masks. Furthermore, the cloth can be used for blood purification by removing toxins and drugs[25].

7.3.2 Defence

Facelets used to protect personnel from harmful gases and vapours are often rigid and clumsy when fitted with granular charcoal, but this problem can be overcome if charcoal cloth is used. Charcoal cloth also has a distinct advantage over other types of charcoal in protective clothing in that it allows more freedom of movement when working in confined contaminated spaces. It is also claimed that charcoal cloth provides gas warfare protective suits with an extended shelf life[25].

Filters are used in closed air-conditioning systems such as in aircraft, tanks and submarines. Weight is frequently a premium in their design, therefore, once more charcoal cloth would be advantageous over other carbon filters.

7.3.3 Industrial

As in defensive applications, the charcoal cloth has industrial uses in respiratory devices and protective clothing for persons working in contaminated environments. Moreover, since charcoal cloth possesses excellent insulating properties, it can be used in fire and heat resisting garments. Small sections of charcoal cloth have already proved useful in lapel badges for analysis of workers' exposure to toxic atmospheres[23].

Charcoal cloth can also be used in industrial air conditioning systems[25], particularly in food processing plants, offices and in the laboratory. In fact, the potential of charcoal cloth in fume hoods is, at present, being investigated by Podder[96].

Wilkins[32] developed a continuous system for removing trace organics from water and results of his work indicated that, at steady state, 90% phenol and 98% 2-chlorophenol were removed from water. This was between three and four times greater than for granular charcoal. Bearing this evidence in mind, it is believed that charcoal cloth will enhance the treatment of industrial and municipal waste water. Furthermore, it is anticipated that charcoal cloth will demonstrate its usefulness for removing colours and dyes from dry cleaning solvents, purifying wines and refining sugar solutions.

Activated charcoal has numerous catalytic applications[97]. For example, it is used as a carrier for aluminium chloride catalyst and as a catalyst itself in the reaction of steam and chlorine to form hydrogen chloride. Many more examples of the catalyst activities of carbon are known and there are thus many possible areas in catalytic systems in which the charcoal cloth might be used. However, it is expected that a considerable amount of work must be undertaken before the viability of the catalytic applications of charcoal cloth are fully determined.

As mentioned in Section 7.2, it is envisaged that activated charcoal cloth might be employed in adsorbing radio-active particles, thus protecting the environment from radio-active emissions.

7.3.4 Domestic

It is contemplated that activated charcoal cloth will have several applications in the home. It could be used, for example, in removing objectionable odours which might arise from cooking, smoking and the metabolism of the occupants, particularly if there is little or no ventilation. Similarly, the charcoal cloth has a potential use as an air purifier in motor vehicles.

The cost of charcoal cloth for domestic purposes would not be excessive since the quantities of the material used would be relatively small.

7.3.5 Combating Static Electricity

Static electricity can be a hazard in the chemical industry and has been the cause of a number of accidents[98]. For example, static electricity can be generated by a pulverized material being discharged through a chute, in a pipeline through which wet steam or a vapour stream containing particulate matter is flowing, on a conveyor belt in motion and the relative motion of two contracting surfaces at least one of which is a poor electrical conductor[99].

As the electric charge accumulates, the electric field strength increases and may do so to such an extent that the air becomes ionized and a spark is generated. In an explosive environment, this of course has to be avoided. One solution is to prevent the accumulation of a charge and this can be achieved by using conductive material. The physical state of charcoal makes it a very versatile material in that it can be simply wrapped around or attached to non-conducting surfaces.

Similarly, 'static' which may exist whilst playing records could be removed by placing a layer of charcoal cloth on the record turntable.

7.4 Conclusion

In spite of evidence inferring that granular charcoal would be more feasible than charcoal cloth for industrial solvent recovery plants, there is still a great market potential for charcoal cloth in numerous other fields.

One of the major assets of charcoal cloth lies in its physical state in that it is very flexible. It can be easily handled and as well as being a better adsorbent per unit mass than granular charcoals, it also has excellent thermal insulating properties.

It is expected that charcoal cloth will become increasingly competitive in the activated carbon market as its cost falls with increased production. As legislation with respect to controlling the discharge of pollutants into water and the atmosphere becomes more stringent, it is anticipated that the best available technology in achieving this would be met by a system using charcoal cloth.

CHAPTER 8

CONCLUSION AND RECOMMENDATIONS

8 CONCLUSION AND RECOMMENDATIONS

The objectives of the research programme may be divided into six general areas.

(i) To assess the adsorption capacity of charcoal cloth, establishing the extent it could remove valuable vapour phase solvents from air in a dynamic batch situation.

(ii) To study the potential of the cloth to give up the solvent materials during regeneration using steam.

(iii) To investigate any reduction in cloth activity for an increasing number of regenerations.

(iv) To develop a mathematical model to predict the performance of the charcoal cloth in a batch process for scale-up purposes.

(v) The physical construction and commissioning of experimental apparatus to investigate the possibility of continuous recovery of vapour phase solvents.

(vi) To identify possible market areas into which charcoal cloth would have useful applications.

The ability of fresh charcoal cloth to take up perchloroethylene and toluene vapours from air under dynamic conditions was studied. The charcoal cloth generally weighing 0.045kg and measuring 0.72m x 0.60m was wound on a wooden former and placed in a cylinder through which the air/vapour mixture was passed. The uptake of solvent per unit mass of cloth varied between 0.319 $kg.kg^{-1}$ and 0.388 $kg.kg^{-1}$ for toluene and 0.732 $kg.kg^{-1}$ increasing to 0.824 $kg.kg^{-1}$ in the case of perchloroethylene vapour. The equilibrium data shown in Figures 4.4 and 4.8 for the chlorinated solvent and toluene respectively resemble Langmuir isotherms[57], suggesting monolayer adsorption.

The adsorption waves, plots of the exit solvent concentration in the air against adsorption time, are presented in Figures 4.5 and 4.6 for perchloroethylene and Figures 4.9 and 4.10 for toluene.

Doulah[95] proposed that the behaviour of the adsorption wave could be expressed by

$$\ln\left(1 - \frac{y}{y_o}\right) = -\left(\frac{t}{\gamma}\right)^{\beta}$$

where the constant γ corresponds to a time when 63% of the inlet concentration of the solvent is registered at the adsorber outlet and β is related to the shape of the adsorption wave.

This relationship was tested using the experimental data obtained from the batch operations and, as presented in Chapter 5, does appear to hold true. The constants evaluated for each of the experimental conditions generally infer that mass transfer coefficient is proportional to solvent concentration and superficial gas velocity.

The relationship derived by Collins[79], used to express the length of unused bed at breakthrough, was also tested. However, it was considered invalid for some of the operations undertaken on the batch set-up. The main failure was thought to occur at high solvent concentrations when small breakthrough times were recorded, leading to long mass transfer zones. The equation proposed by Collins assumes a small adsorption zone height in relation to the height of the adsorption bed. Since relationships have been formulated to express the cloth's equilibrium capacities and the adsorption waves, then its performance can be predicted for scale up purposes.

Some indication of the optimum adsorption conditions can be obtained from experimental data quoted in Chapters 2 and 4. The laboratory solvent uptake measurements taken at CDE Porton Down clearly show that the cloth's adsorption capacity is enhanced under dry conditions. It is pointed out in Section 4.4 that for a given temperature and humidity, the most favourable results on the batch rig were achieved at low superficial gas velocities and at low solvent vapour concentrations.

The charcoal cloth has a greater surface area per unit mass than nutshell carbons which suggests its higher adsorption capacities. The larger surface areas are attributed to the slit shaped micro pores situated at the surface of the cloth, as illustrated in Figure 4.14. It is believed that this charcoal cloth property is the reason for the high volumes of solvent recovered for relatively small quantities of steam during the regeneration process. Since the adsorbed molecules were held in the short micro pores which are directly exposed to the outer surface of the cloth, the resistances to heat and mass transfer are regarded as small relative to those assumed for desorption from the complex network of macro, transitional and micro pores inherent in nutshell active carbons.

Furthermore, the nature of the porous structure of activated charcoal cloth is also attributed to the comparatively high quantities of organic solvent recovered after two, three, four and five adsorption-desorption cycles. The amount of solvent recovered expressed as a percentage of the weight of virgin cloth was found to be 23.26% and 56.73% for toluene and perchloroethylene respectively after the first cycle. The average values computed for subsequent cycles were 24.20% for toluene and 45.83% for perchloroethylene.

Smith[100] analysed regenerated cloth samples from this work and investigated for any reduction in charcoal cloth activity with increasing number of regenerations. His measurements on charcoal cloth which had been used to adsorb perchloroethylene vapour showed that there was an immediate drop in the cloth's activity after one regeneration, but then remained fairly constant with an increasing number of regenerations. It was also found that at least 30% of the charcoal cloth activity remained after 15 regenerations, and that the reduction in residual activity of the cloth was not as great when involved in taking up toluene vapour.

The solvent vapour uptake tests carried out at CDE Porton Down clearly show that the charcoal cloth is more favourable than nutshell charcoal. However, a somewhat different conclusion is drawn when comparing the two types of active carbon for a solvent recovery process. At present, the cost of unit mass of granular charcoal is less than one per cent of that for activated charcoal cloth. Additionally, the capital expenditure requirements for an adsorber unit would be much higher for the cloth because of its relatively low bulk density. The pressure drop across the adsorbent bed would not be acceptable if the cloth were wound round an annulus or packed in discs so as to give a high bulk density. Much greater quantities of the cloth would have to be produced so as to lower significantly its cost before charcoal cloth can be regarded as a serious competitor to granular charcoals on batchwise solvent recovery plants in spite of its greater adsorptive capacity and low steam requirements for regeneration.

Experimental equipment was constructed to assess the charcoal cloth's performance for the continuous recovery of valuable vapour phase solvents, and this is described in Section 6.1. The results, however, showed that at best, 71% (by volume) of the perchloroethylene vapour was adsorbed, but generally values between 45 and 55% were obtained. In order to improve the performance of the continuous system, it is recommended that a much longer adsorber duct be used and that it be fitted with deflector baffles to direct the vapour mixture onto the cloth.

Although solvent was being desorbed, none was actually recovered due to ingress of air into the regenerator. The whole system should be completely sealed from the atmosphere if the organic solvents are to be successfully recovered. In spite of these recommendations, replacing the cloth is laborious. Bearing this in mind, an alternative continuous solvent recovery unit has been suggested whereby the charcoal cloth wound on reels is transported across the width of the duct, perpendicular to the flowing gas stream. The system is depicted in Figure 6.5.

The potential of activated charcoal cloth on industrial solvent recovery plants, at present, does not appear to be viable. The cost of the cloth needs to fall considerably before it can seriously be considered for the batchwise recovery of valuable vapour phase solvents. Further modifications to the continuous set-up and an improvement in the analytical techniques are needed before the continuous reclamation of organic solvent materials can be fully assessed.

However, Chapter 7 highlights some of the market areas where charcoal cloth would have useful applications. The flexibility of its physical state, high surface area per unit mass, thermal insulating properties and its potential to conduct electricity can be exploited in numerous industrial, defence, medical and domestic applications.

NOMENCLATURE

a_c	air flow resistance of charcoal cloth	$(m.water\ gauge.m^{-1}.s^{-1})$
c	constant in Henry's Law equation	
D	height of adsorbent bed	(m)
D_a	height of adsorbent zone	(m)
D_e	effective diffusivity	$(m^2.s^{-1})$
D_k	Knusden diffusivity	$(m^2.s^{-1})$
f	fractional ability of adsorption zone to adsorb solute	
F	free energy	$(J.kg^{-1})$
G_s	mass velocity of unadsorbed gas based on column cross section	$(kg.m^{-2}.s^{-1})$
H	enthalpy	$(J.kg^{-1})$
$\overline{H}$	differential heat of adsorption	$(J.kg^{-1})$
ΔH_a	integral heat of adsorption referred to liquid adsorbate per unit mass of adsorbent	$(J.kg^{-1})$
$\Delta H_a'$	integral heat of adsorption referred to vapour adsorbate per unit mass of adsorbent	$(J.kg^{-1})$
k	constant in Langmuir isotherm equation	
k'	constant in Freundlich equation	
k"	constant in Brunauer, Emett and Teller equation	
k_c	external gas film mass transfer coefficient	$(moles.m^{-2}.s^{-1})$
K_{OG}	overall mass transfer coefficient	$(moles.m^{-2}.s^{-1})$
L	length of unused adsorbent bed	(m)
M	molecular weight	
n	constant in Freundlich equation	
p	equilibrium partial pressure of solute in solvent in vapour phase	
p^o	vapour pressure of solute vapour	

Δp	pressure drop across the adsorbent bed	(m.water gauge)
r	radial parameter	(m)
r_p	pore radius	(m)
R	gas constant	
S	entropy	(J.kg^{-1}.k^{-1})
t	time	(s)
t_B	time to breakthrough	(s)
t_e	time to attain equilibrium	(s)
t_s	time to idealized breakthrough	(s)
T	absolute temperature	(K)
$\overline{U}$	mean molecular velocity	(m.s^{-1})
V	volume of gas adsorbed at STP per unit mass of adsorbent	(m^3)
V_m	volume of gas adsorbed at STP per unit mass of adsorbent with layer one molecule thick	(m^3)
w_c	weight of charcoal cloth	(kg)
W	quantity of gas adsorbed per unit mass of solid adsorbent	
W_e	adsorbate concentration, mass solute per mass adsorbent at equilibrium	(kg.kg^{-1})
W_o	initial adsorbate concentration, mass solute per mass adsorbent	(kg.kg^{-1})
x	$= p/p_o$	
y	gas phase concentration	(moles.m^{-3})
y_B	effluent gas phase concentration at break-point	(moles.m^{-3})
y_e	equilibrium gas phase concentration	(moles.m^{-3})
y_o	initial gas phase concentration	(moles.m^{-3})
y_p	gas phase concentration in pore	(moles.m^{-3})
y_s	effluent gas phase concentration, virtually at equilibrium	(moles.m^{-3})
Y_o	initial concentration of solute in gas phase, mass solute per mass non-adsorbing gas	(kg.kg^{-1})

Y_o^*	initial concentration of solute in gas phase in equilibrium	$(kg.kg^{-1})$
β	constant related to shape of adsorption wave	
γ	constant, inversely proportion to K_{OG}	
Δ	difference	
$\int$	degree of saturation of adsorbent bed at breakpoint	
λ	latent heat of vapourization	$(J.kg^{-1})$
ρ_c	bulk density of charcoal cloth	$(kg.m^{-3})$
ρ_s	apparent density of adsorbent bed	$(kg.m^{-3})$

Subscripts

B	at breakpoint
c	charcoal cloth
e	at equilibrium
o	initial
r	reference substance

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APPENDIX I

DETERMINATION OF PRESSURE DROP ACROSS A PACKED BED OF CHARCOAL CLOTH DISCS AT A SUPERFICIAL LINEAR GAS VELOCITY OF 0.134 MIS

DATA

Bulk density of charcoal cloth, ρ_c = $2.5 - 3.0 \times 10^2$ kg/m^3 (under a load of 5×10^2 kg/m^2)

Air flow resistance per layer or disc of cloth, a_c = $0.7 - 1.2 \times 10^{-2}$m water gauge per m/s air

Height of charcoal cloth bed, D = 0.60m

Radius of bed = radius of cloth disc, r = 1.905×10^{-2}m

Weight of 1m of cloth, w_c = 0.110 kg

Volume occupied by charcoal cloth bed

$$= \pi r^2 D$$
$$= \pi(1.905 \times 10^{-2})^2 \ (0.6) \ \text{m}^3$$
$$= \underline{6.84 \times 10^{-4} \ \text{m}^3}$$

Assuming ρ_c = 3 x 10 kg/m , the total weight of the charcoal cloth bed

$$= (3 \times 10^2)(6.84 \times 10^{-4}) \ \text{kg}$$
$$= \underline{0.2052 \ \text{kg}}$$

Area of each disc of cloth

$$= \pi r^2$$
$$= \pi(1.905 \times 10^{-2})^2 \ \text{m}^2$$
$$= \underline{1.14 \times 10^{-3} \ \text{m}^2}$$

Since 1m^2 of cloth weighs 0.110kg, then the weight of 1 disc of cloth

$$= (1.14 \times 10^{-3})(0.110)$$
$$= \underline{1.254 \times 10^{-4} \ \text{kg}}$$

Therefore, number of cloth discs making up the bed

$$= \frac{0.2052}{1.254 \times 10^{-4}}$$
$$= \underline{1.636 \times 10^3}$$

Assuming an air flow resistance of 1.2×10^{-2}m water per m/s air, the pressure drop across the whole bed at a linear air velocity of 0.134 m/s

$$\Delta P = (1.2 \times 10^{-2})(1.636 \times 10^3)(0.134) \text{m water gauge}$$
$$\Delta P = \underline{2.63\text{m water gauge}}$$

Since the bed height is 0.60m, the pressure drop per 0.1m bed

$$\Delta P = \frac{2.63}{6} \ \text{m water gauge}$$
$$\Delta P = \underline{0.44\text{m water gauge}}$$

APPENDIX II

TABLES OF THE EXPERIMENTAL CONDITIONS, AND THE ADSORPTION, REGENERATION AND CONDENSATE RECOVERY RESULTS WITH REGARD TO THE BATCH OPERATIONS

Expt. No.	Liquid Solvent Rate ($m^3.s^{-1}$) (x 10^8)	Air Rate ($m^3.s^{-1}$) (x 10^4)	Volumetric Concentration (vol. %)	$\frac{\text{Wt. Solvent}}{\text{Wt. Air}}$ ($kg.kg^{-1}$)	Superficial Gas Velocity ($m.s^{-1}$)	Temperature (K)	Relative Air Humidity (%)	Column Pressure Drop (mH_2O)
BP1	0.25	1.542	0.375	0.0215	0.1356	294.8	46.0	0.018
BP2	0.33	1.543	0.500	0.0286	0.1360	295.0	51.0	0.011
BP3	0.50	1.532	0.751	0.0429	0.1354	295.4	53.0	0.017
BP4	0.66	1.575	1.000	0.0574	0.1395	295.3	48.0	0.035
BP5	1.00	1.548	1.500	0.0859	0.1378	294.7	48.0	0.019
BP6	1.33	1.543	1.998	0.1145	0.1349	295.3	42.0	0.015
BP7	0.25	1.572	0.374	0.0215	0.1384	291.3	47.5	0.020
BP8	0.33	1.551	0.500	0.0286	0.1362	295.3	48.0	0.017
BP9	0.50	1.553	0.749	0.0430	0.1363	295.5	49.0	0.010
BP10	0.66	1.551	1.000	0.0573	0.1362	294.2	46.5	0.017
BP11	1.33	1.582	2.000	0.1146	0.1390	295.9	48.8	0.015
BP12	0.25	0.501	1.186	0.0681	0.0400	296.2	55.0	0.007
BP13	0.50	0.984	1.216	0.0697	0.0864	294.5	54.3	0.008
BP14	0.66	1.284	1.242	0.0700	0.1127	294.8	56.0	0.022

TABLE 1: EXPERIMENTAL CONDITIONS FOR THE ADSORPTION OF PERCHLOROETHYLENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Weight of Fresh Cloth (kg) (x 10^2)	Weight of Cloth at Equilibrium (kg) (x 10^2)	Weight of Solvent Adsorbed (kg) (x 10^2)	Weight of Solvent Adsorbed per Unit Cloth Weight ($kg.kg^{-1}$)	Time to Attain Equilibrium (secs) (x 10^4)
BP1	4.437	7.778	3.341	0.753	2.754
BP2	4.497	7.791	3.294	0.732	1.632
BP3	4.477	7.782	3.305	0.738	1.134
BP4	5.098	8.905	3.807	0.747	0.648
BP5	4.507	7.897	3.390	0.752	0.360
BP6	4.484	7.905	3.421	0.763	0.282
BP7	4.582	8.082	3.500	0.764	2.070
BP8	4.706	8.399	3.693	0.784	2.022
BP9	4.410	7.634	3.224	0.731	1.476
BP10	4.491	8.218	3.727	0.830	1.272
BP11	4.480	8.170	3.690	0.824	0.564
BP12	4.512	8.202	3.690	0.818	2.472
BP13	4.522	8.819	3.667	0.811	1.266
BP14	4.504	8.097	3.575	0.794	1.158

TABLE 2: ADSORPTION RESULTS FOR THE UPTAKE OF PERCHLOROETHYLENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Total Time for Steaming (secs)	Time to Heat up Equipment (secs)	Time Condensate Collected (secs)	Total Mass of Steam Used (kg) ($x 10^2$)	Total Mass of Solvent Recovered (kg) ($x 10^2$)	Percent. of Solvent Recovered to that Adsorbed (%)	Overall Steam -Solvent Ratio ($kg.kg^{-1}$)
BP1	840	300	540	15.540	1.948	58.30	7.98
BP2	453	273	180	8.380	1.850	56.19	4.53
BP3	823	283	540	15.225	1.915	57.94	7.95
BP4	815	275	540	15.077	2.597	68.21	5.81
BP5	800	260	540	14.800	2.175	64.16	6.80
BP6	665	305	360	12.302	1.623	47.44	7.58
BP7	505	325	180	9.342	1.866	53.33	5.01
BP8	618	258	360	11.433	2.175	58.90	5.26
BP9	624	264	360	11.544	1.607	49.84	7.18
BP10	459	279	180	8.491	1.980	53.13	4.29
BP11	477	297	360	8.824	2.061	55.85	4.28
BP12	645	285	360	11.932	1.704	46.18	7.00
BP13	462	282	180	8.547	1.331	36.30	6.42
BP14	446	266	180	8.251	1.753	49.03	4.71

TABLE 3: REGENERATION RESULTS FOR THE DESORPTION OF PERCHLOROETHYLENE FROM ACTIVATED CHARCOAL CLOTH

			1st Time Period		2nd Time Period		Cumulative after 2nd Time Period		3rd Time Period		Cumulative after 3rd Time Period	
Expt. No.	Time Condensate Collected (secs)	Time Intervals (secs)	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio (kg.kg^{-1})	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio (kg.kg^{-1})	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio (kg.kg^{-1})	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio (kg.kg^{-1})	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio (kg.kg^{-1})
BP1	540	180	34.98	1.01	16.52	4.82	51.50	2.23	6.80	12.06	58.30	6.90
BP2	180	60	33.53	0.18	14.78	1.56	48.31	0.60	7.88	3.54	56.19	1.01
BP3	540	180	39.28	0.67	12.76	4.83	52.04	1.69	5.89	11.96	57.94	6.23
BP4	540	180	55.42	0.76	8.53	9.24	63.95	1.90	4.26	19.10	68.21	5.17
BP5	540	180	47.88	1.11	10.53	7.28	58.41	2.22	5.74	15.04	64.16	6.20
BP6	360	180	28.46	2.26	18.98	3.82	47.44	2.89	-	-	-	-
BP7	180	60	28.29	0.32	19.48	1.00	47.77	0.66	5.56	4.00	53.33	1.55
BP8	360	120	45.71	0.89	8.79	6.41	54.50	1.78	4.40	13.43	58.90	2.65
BP9	360	120	31.21	1.53	13.09	5.43	44.30	2.68	5.54	12.66	49.84	3.79
BP10	180	60	29.18	0.42	16.98	0.95	46.16	0.62	6.97	4.54	53.13	1.13
BP11	360	120	36.07	0.59	14.51	2.24	50.58	1.06	5.27	7.60	55.85	1.68
BP12	360	120	32.55	0.99	9.67	6.05	42.22	2.15	3.95	13.49	46.18	3.12
BP13	180	60	15.05	0.54	12.39	1.36	27.44	0.91	8.86	1.97	36.30	1.17
BP14	180	60	28.15	0.19	14.07	1.23	42.22	0.54	6.81	3.70	49.03	0.98

TABLE 4: CONDENSATE RECOVERY RESULTS FOR THE DESORPTION OF PERCHLOROETHYLENE FROM ACTIVATED CHARCOAL CLOTH

Expt. No.	Liquid Solvent Rate ($m^3.s^{-1}$) ($x\ 10^8$)	Air Rate ($m^3.s^{-1}$) ($x\ 10^4$)	Volumetric Concentration (vol %)	$\frac{\text{Wt. Solvent}}{\text{Wt. Air}}$ ($kg.kg^{-1}$)	Superficial Gas Velocity ($m.s^{-1}$)	Temperature (K)	Relative Air Humidity (%)	Column Pressure Drop (mH_2O)
BT1	0.25	1.551	0.360	0.0115	0.1361	294.7	46.4	0.022
BT2	0.33	1.523	0.480	0.0153	0.1336	294.5	48.0	0.015
BT3	0.50	1.584	0.721	0.0229	0.1390	293.7	46.0	0.020
BT4	0.50	1.596	0.720	0.0229	0.1400	295.2	46.5	0.022
BT5	0.66	1.553	0.961	0.0306	0.1362	294.2	47.0	0.033
BT6	1.00	1.526	1.442	0.0458	0.1339	295.1	48.7	0.017
BT7	1.33	1.571	1.921	0.0611	0.1378	294.6	48.0	0.015
BT8	0.25	0.500	1.140	0.0363	0.0439	294.3	59.0	0.006
BT10	0.50	0.943	1.190	0.0378	0.0827	293.9	47.0	0.008
BT11	0.66	1.267	1.185	0.0377	0.1111	294.5	48.0	0.017

TABLE 5: EXPERIMENTAL CONDITIONS FOR THE ADSORPTION OF TOLUENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Weight of Fresh Cloth (kg) (x 10^2)	Weight of Cloth at Equilibrium (kg) (x 10^2)	Weight of Solvent Adsorbed (kg) (x 10^2)	Weight of Solvent Adsorbed per Unit Cloth Weight ($kg.kg^{-1}$)	Time to Attain Equilibrium (secs) (x 10^4)
BT1	4.618	6.093	1.475	0.319	2.520
BT2	4.305	5.800	1.495	0.347	1.896
BT3	4.663	6.215	1.552	0.333	1.752
BT4	4.563	6.113	1.567	0.345	1.440
BT5	4.458	6.110	1.652	0.371	1.320
BT6	4.347	6.016	1.667	0.384	0.810
BT7	4.396	6.100	1.704	0.388	0.708
BT8	4.794	6.520	1.726	0.360	2.634
BT10	4.423	6.060	1.637	0.370	1.548
BT11	4.694	6.341	1.647	0.351	0.954

TABLE 6: ADSORPTION RESULTS FOR THE UPTAKE OF TOLUENE VAPOUR ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Total Time for Steaming (secs)	Time to Heat Up Equipment (secs)	Time Condensate Collected (secs)	Total Mass of Steam Used (kg) ($x\ 10^2$)	Total Mass of Solvent Recovered (kg) ($x\ 10^2$)	% of Solvent Recovered to that Adsorbed (%)	Overall Steam Solvent Ratio ($kg.kg^{-1}$)
BT1	455	275	180	8.417	0.675	45.76	12.47
BT2	440	260	180	8.140	0.649	43.41	12.54
BT3	485	305	180	8.972	0.589	37.95	15.23
BT4	614	254	360	11.359	0.883	56.35	12.86
BT5	441	261	180	8.158	0.979	59.26	8.33
BT6	825	285	540	15.262	1.368	82.06	11.15
BT7	453	273	180	8.380	0.900	52.86	9.31
BT8	456	276	180	8.436	0.909	52.68	9.28
BT9	690	330	360	12.765	0.883	53.94	14.45
BT10	648	288	360	11.988	0.961	58.35	12.47

TABLE 7: REGENERATION RESULTS FOR THE DESORPTION OF TOLUENE FROM ACTIVATED CHARCOAL CLOTH

			1st Time Period		2nd Time Period		Cumulative after 2nd Time Period		3rd Time Period		Cumulative after 3rd Time Period	
Expt. No.	Time Condensate Collected (secs)	Time Intervals (secs)	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio $(kg.kg^{-1})$	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio $(kg.kg^{-1})$	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio $(kg.kg^{-1})$	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio $(kg.kg^{-1})$	% of Solvent Recovered to that Adsorbed (%)	Steam Solvent Ratio $(kg.kg^{-1})$
BT1	180	60	34.64	0.47	8.81	5.39	43.45	1.47	2.35	28.87	45.80	2.49
BT2	180	60	14.48	1.84	22.00	2.43	36.49	1.90	6.94	8.26	43.41	3.20
BT3	180	60	2.79	10.85	24.00	1.69	26.79	2.64	11.16	5.66	37.95	3.53
BT4	360	120	40.90	1.78	9.94	13.60	50.84	4.09	5.51	22.51	56.35	5.90
BT5	180	60	43.00	0.68	11.01	4.78	54.01	1.67	5.25	12.00	59.26	2.44
BT6	540	180	57.08	1.57	20.74	6.35	77.82	2.85	4.14	56.58	82.06	5.57
BT7	180	60	30.00	0.57	14.22	2.56	44.22	1.21	8.64	4.82	52.86	1.80
BT8	180	60	23.08	1.08	16.06	2.24	39.14	1.55	13.54	4.98	52.68	2.43
BT9	360	120	35.44	1.26	14.81	3.88	50.24	2.03	3.70	21.94	53.94	3.40
BT10	360	120	35.23	1.62	15.26	6.05	50.48	2.96	7.89	13.93	58.35	4.44

TABLE 8: CONDENSATE RECOVERY RESULTS FOR THE DESORPTION OF TOLUENE FROM ACTIVATED CHARCOAL CLOTH

Expt. No.	Liquid Solvent Rate $(m.s^{-1})$ $(\times 10^8)$	Air Rate $(m^3.s^{-1})$ $(\times 10^4)$	Volumetric Concentration (vol %)	$\frac{\text{Wt. Solvent}}{\text{Wt. Air}}$ $(kg.kg^{-1})$	Superficial Gas Velocity $(m.s^{-1})$	Temperature (K)	Relative Air Humidity (%)	Column Pressure Drop (mH_2O)
CBP1	1.00	1.574	1.499	0.0859	0.1383	295.5	48.0	0.015
CBP2	1.00	1.565	1.502	0.0859	0.1375	295.4	48.0	0.016
CBP3	1.00	1.566	1.500	0.0859	0.1376	295.0	44.9	0.018
CBP4	1.00	1.563	1.500	0.0859	0.1373	295.1	47.9	0.020
CBT1	1.00	1.552	1.440	0.0459	0.1363	296.2	49.5	0.017
CBT2	1.00	1.548	1.441	0.0459	0.1360	296.6	47.6	0.015
CBT3	1.00	1.528	1.441	0.0459	0.1343	296.1	49.0	0.013
CBT4	1.00	1.526	1.442	0.0459	0.1341	296.0	49.0	0.020
CBT5	1.00	1.528	1.441	0.0459	0.1343	296.4	47.)	0.012

TABLE 9: CONDITIONS FOR THE CYCLIC ADSORPTION-DESORPTION OPERATIONS FOR THE UPTAKE OF PERCHLOROETHYLENE AND TOLUENE VAPOURS ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Wt.of Virgin Cloth (kg) $(x10^2)$	Wt. of Cloth at Start of a Run (kg) $(x\ 10^2)$	Wt. of Material Adsorbed at the Start of a Run (kg) $(x\ 10^2)$	Wt. of Cloth at Equilibrium (kg) $(x\ 10^2)$	Total Wt. of Material Adsorbed (kg) $(x\ 10^2)$	Wt. of Solvent Taken Up During a Run (kg) $(x\ 10^2)$	Ratio of Solvent Taken Up During a Run and Wt. of Virgin Cloth $(kg.kg^{-1})$ $(x\ 10^2)$	Ratio of Wt. of Cloth at Equilibrium and Wt. of Virgin Cloth $(kg.kg^{-1})$ $(x\ 10^2)$	Time to Attain Equilibrium (secs) $(x\ 10^4)$
CBP1	4.520	4.520	-	8.210	3.690	3.690	0.816	0.816	1.254
CBP2	4.520	5.713	1.193	8.193	3.673	2.480	0.548	0.813	0.828
CBP3	4.520	5.613	1.093	8.028	3.508	2.415	0.534	0.776	0.660
CBP4	4.520	5.977	1.457	8.065	3.543	2.088	0.462	0.784	0.504
CBT1	4.617	4.617	-	6.267	1.650	1.650	0.357	0.357	1.020
CBT2	4.617	5.522	0.905	6.305	1.688	0.783	0.170	0.365	0.672
CBT3	4.617	5.087	0.470	6.224	1.607	1.137	0.246	0.348	0.888
CBT4	4.617	5.737	1.120	6.235	1.618	0.498	0.108	0.350	0.816
CBT5	4.617	5.840	1.223	6.217	1.600	0.377	0.082	0.347	0.918

TABLE 10: ADSORPTION RESULTS FOR THE CYCLIC ADSORPTION-DESORPTION OPERATIONS FOR THE UPTAKE OF PERCHLOROETHYLENE AND TOLUENE VAPOURS ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Total Time for Steaming (secs)	Time to Heat up Equipment (secs)	Time Condensate Collected (secs)	Total Mass of Steam Used (kg) (x 10^2)	Total Mass of Solvent Recovered (kg) (x 10^2)	Percent. of Solvent Recovered to Mass of Virgin Cloth (%)	Overall Steam -Solvent Ratio ($kg.kg^{-1}$)
CBP1	637	277	360	11.784	2.564	56.73	4.60
CBP2	600	240	360	11.100	2.061	45.60	5.38
CBP3	623	263	360	11.525	2.288	50.62	5.04
CBP4	607	247	360	11.230	1.866	41.28	6.02
CBT1	640	280	360	11.840	1.074	23.26	11.02
CBT2	606	246	360	11.211	1.186	25.69	9.45
CBT3	617	257	360	11.414	1.108	24.00	10.30
CBT4	634	274	360	11.729	1.091	23.63	10.75
CBT5	623	263	360	11.525	1.083	23.46	10.64

TABLE 11: REGENERATION RESULTS WITH REGARD TO THE CYCLIC ADSORPTION-DESORPTION OPERATIONS FOR THE DESORPTION OF PERCHLOROETHYLENE AND TOLUENE FROM ACTIVATED CHARCOAL CLOTH

			1st Time Period		2nd Time Period		Cumulative after 2nd Time Period		3rd Time Period		Cumulative after 3rd Time Period	
Expt. No.	Time Condensate Collected (secs)	Time Intervals (secs)	% of Solvent Recovered to Mass of Virgin Cloth (%)	Steam Solvent Ratio ($kg.kg^{-1}$)	% of Solvent Recovered to Mass of Virgin Cloth (%)	Steam Solvent Ratio ($kg.kg^{-1}$)	% of Solvent Recovered to Mass of Virgin Cloth (%)	Steam Solvent Ratio ($kg.kg^{-1}$)	% of Solvent Recovered to Mass of Virgin Cloth (%)	Steam Solvent Ratio ($kg.kg^{-1}$)	% of Solvent Recovered to Mass of Virgin Cloth (%)	Steam Solvent Ratio ($kg.kg^{-1}$)
CBP1	360	120	43.45	0.48	12.20	4.24	55.65	1.30	1.08	34.71	56.73	1.94
CBP2	360	120	22.26	1.17	15.80	1.47	38.06	1.30	7.54	5.40	45.60	1.97
CBP3	360	120	39.14	0.67	7.54	6.51	46.68	1.62	3.94	12.71	50.62	2.48
CBP4	360	120	23.70	1.29	11.85	3.29	35.55	1.95	5.73	12.42	41.28	2.91
CBT1	360	120	17.07	0.99	4.87	7.68	21.94	2.48	1.32	31.17	23.26	4.09
CBT2	360	120	10.88	1.77	11.06	3.54	21.94	2.66	3.75	11.95	25.69	4.02
CBT3	360	120	18.57	1.72	3.56	9.42	21.13	2.96	2.87	19.98	24.00	4.29
CBT4	360	120	17.44	1.19	4.31	8.99	21.75	2.73	1.88	22.50	23.63	4.31
CBT5	360	120	16.32	2.22	5.25	10.02	21.57	3.56	1.89	27.48	23.46	5.99

TABLE 12: CONDENSATE RECOVERY RESULTS WITH REGARD TO CYCLIC ADSORPTION-DESORPTION OPERATIONS

APPENDIX III

DATA USED TO TEST THE APPLICATION OF THE LANGMUIR AND FREUNDLICH ISOTHERMS FOR THE ADSORPTION OF PERCHLOROETHYLENE AND TOLUENE VAPOURS ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Mass of Perchloroethylene Adsorbed per Unit Mass of Cloth	Partial Pressure of Perchloroethylene in Air			Volume of Perchloroethylene Adsorbed at STP per Unit Mass of Cloth		
	W $(kg.kg^{-1})$	P (atm)	$\frac{1}{P}$ (atm^{-1})	In P	V (m^3)	$\frac{1}{V}$ (m^{-3})	In V
BP2	0.732	0.00497	201.0	-5.304	0.0989	10.113	-2.314
BP3	0.738	0.00726	137.7	-4.925	0.0996	10.040	-2.307
BP4	0.747	0.00958	104.4	-4.648	0.1010	9.901	-2.293
BP5	0.752	0.01486	67.3	-4.209	0.1016	9.843	-2.287
BP6	0.763	0.01976	50.6	-3.924	0.1031	9.700	-2.272
BP7	0.764	0.00370	270.1	-5.599	0.1033	9.682	-2.270
BP1	0.753	0.00378	264.6	-5.578	0.1018	9.821	-2.285
BP8	0.784	0.00492	203.0	-5.314	0.1060	9.443	-2.244
BP10	0.830	0.00998	100.2	-4.607	0.1121	8.917	-2.188
	0.816	0.01453	68.8	-4.231	0.1113	8.984	-2.195
BP11	0.824	0.01988	50.3	-3.918	0.1103	9.066	-2.204

TABLE 1: SUMMARY OF DATA USED TO TEST THE APPLICATION OF LANGMUIR AND FREUNDLICH ISOTHERMS FOR THE ADSORPTION OF PERCHLOROETHYLENE ONTO ACTIVATED CHARCOAL CLOTH

Expt. No.	Mass of Toluene Adsorbed per Unit Mass of Cloth	Partial Pressure of Toluene in Air			Volume of Toluene Adsorbed at STP per Unit Mass of Cloth		
	W $(kg.kg^{-1})$	P (atm)	$\frac{1}{P}$ (atm^{-1})	In P	V (m^3)	$\frac{1}{V}$ (m^{-3})	In V
BT1	0.319	0.00365	274.0	-5.613	0.0776	12.887	-2.556
BT2	0.347	0.00497	201.2	-5.304	0.0844	11.848	-2.472
BT3	0.333	0.00715	140.0	-4.941	0.0810	12.346	-2.513
BT4	0.345	0.00715	140.0	-4.941	0.0848	11.791	-2.467
BT5	0.371	0.00980	102.0	-4.625	0.0873	11.455	-2.438
	0.357	0.01496	66.8	-4.202	0.0869	11.513	-2.386
BT6	0.384	0.01500	66.7	-4.200	0.0920	10.871	-2.369
BT7	0.388	0.01940	51.5	-3.942	0.0936	10.683	-2.443

TABLE 2: SUMMARY OF DATA USED TO TEST THE APPLICATION OF LANGMUIR AND FREUNDLICH ISOTHERMS FOR THE ADSORPTION OF TOLUENE ONTO ACTIVATED CHARCOAL CLOTH

APPENDIX IV

TABLES OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t DATA USED IN EVALUATING THE CONSTANTS β AND γ FOR THE VARIOUS EXPERIMENTAL OPERATIONS

Run No:	BP1	Run No:	BP4	Run No:	BP6	Run No:	BP10
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
3.665	-0.7986	3.209	-2.2205	2.778	-0.8857	3.306	-1.7924
3.889	-0.4154	3.477	-0.9462	2.892	-0.5784	3.635	-0.3626
3.930	-0.2917	3.556	-0.4890	3.140	-0.2234	3.718	-0.0821
4.061	-0.0285	3.623	-0.2583	3.255	0.0519	3.799	0.1344
4.124	0.0567	3.681	0.0081	3.346	0.2370	3.908	0.2013
4.233	0.1880	3.732	0.1646	3.924	0.3918	3.945	0.2176
4.268	0.2217	3.778	0.3566	3.450	-	3.993	0.2485
4.308	0.2952	3.791	0.4067	-		4.033	0.2827
4.345	0.3960	3.812	0.5077	-		4.064	0.2751
4.396	0.5669	-	-	-		4.104	0.3097
4.426	0.6838	-	-	-			
4.440	-						

TABLE 1: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - SINGLE ADSORPTION OPERATIONS. EFFECT OF VARYING PERCHLOROETHYLENE VAPOUR CONCENTRATION

Run No: BP12		Run No: BP13		Run No: BP14	
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
3.812	-2.2205	3.431	-1.4118	3.225	-3.8056
4.102	-0.6323	3.653	-1.1866	3.542	-0.9420
4.140	-0.3489	3.737	-0.6712	3.641	-0.0929
4.230	0.0555	3.803	-0.2287	3.723	-0.0917
4.297	0.1763	3.839	-0.0677	3.791	0.0223
4.338	0.2067	3.875	0.0579	3.850	0.1244
4.375	0.2244	3.908	0.1583	3.902	0.1608
-	-	3.940	0.2080	3.948	0.1659
-	-	3.966	0.2485	3.990	0.1880
-	-	4.036	0.2720	4.033	0.2040
-	-	4.061	0.3350	4.064	0.2162
-	-	4.102	0.3511	-	-

TABLE 2: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - SINGLE ADSORPTION OPERATIONS. EFFECT OF VARYING THE SUPERFICIAL GAS VELOCITY OF THE PERCHLOROETHYLENE/AIR MIXTURE

Run No: BT1		Run No: BT5		Run No: BT7	
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
3.791	-1.4742	3.477	-0.9506	2.820	-1.2900
3.857	-1.3675	3.653	-0.2917	3.270	-0.8420
4.043	-1.0305	3.808	-0.0049	3.346	-0.4391
4.100	-0.9638	3.899	0.1724	3.412	0.0093
4.158	-0.5636	3.982	0.3281	3.653	0.1507
4.225	-0.1768	4.045	0.4111	3.742	0.2356
4.255	-0.0176	4.100	0.4736	3.803	0.2705
4.297	0.1841	4.121	0.4915	3.850	0.2781
4.369	0.2528	4.158	0.4915	-	-
4.401	0.3179	-	-	-	-
4.425	0.3622	-	-	-	-

TABLE 3: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - SINGLE ADSORPTION OPERATIONS. EFFECT OF VARYING TOLUENE VAPOUR CONCENTRATION

Run No: BT8		Run No: BT9		Run No: BT10	
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
3.900	-1.9562	3.556	-0.2960	3.057	-2.1533
4.024	-0.4072	3.611	-0.2251	3.577	-0.4477
4.111	-0.3383	3.732	-0.0977	3.692	-0.1467
4.178	-0.2380	3.808	-0.0119	3.787	0.0389
4.225	-0.0034	3.885	0.0758	3.875	0.2484
4.264	0.0710	3.960	0.1469	3.980	0.5577
4.307	0.1567	4.061	0.2189	-	-
4.338	0.2067	4.109	0.2067	-	-
4.366	0.2470	4.153	0.3421	-	-
4.396	0.4045	4.190	-	-	-
4.417	0.4045	-	-	-	-
4.425	0.4045	-	-	-	-

TABLE 4: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - SINGLE ADSORPTION OPERATIONS. EFFECT OF VARYING THE SUPERFICIAL GAS VELOCITY OF THE TOLUENE/AIR MIXTURE

Run No:	CBP1	Run No:	CBP2	Run No:	CBP3	Run No:	CBP4
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
3.193	-2.0952	2.983	-1.3468	3.009	-1.1203	3.057	-0.9866
3.322	-1.2725	3.256	-0.6183	3.283	-0.3550	3.225	-0.4322
3.450	-1.0677	3.412	-0.2327	3.494	0.0116	3.358	-0.1973
3.486	-0.7587	3.502	-0.0546	3.617	0.1772	3.468	-0.0439
3.591	-0.4039	3.584	0.0806	3.697	0.2385	3.549	0.0758
3.670	-0.2611	3.676	0.1134	3.751	0.2067	3.635	0.1685
3.760	0.0531	3.737	0.1789	3.820	0.2385	3.702	0.2067
3.808	0.0854	3.791	0.2203	3.915	0.2616	3.861	0.3315
3.865	0.1344	3.839	0.2370	-	-	-	-
3.912	0.1595	3.878	0.3146	-	-	-	-
4.033	0.2400	3.918	0.3474	-	-	-	-
4.068	0.3213	-	-	-	-	-	-
4.111	0.3622	-	-	-	-	-	-

TABLE 5: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - CYCLIC ADSORPTION OPERATIONS INVOLVING PERCHLOROETHYLENE

Run No: CBT1		Run No: CBT2		Run No: CBT3		Run No: CBT4		Run No: CBT5	
log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$	log.t	log.ln $\frac{y}{yo-y}$
2.954	-1.3368	2.924	-1.3675	2.954	-1.0514	3.079	-1.3468	2.857	-1.2237
3.176	-0.6092	3.158	-0.6161	3.193	-0.5252	3.255	-0.7269	3.057	-0.8972
3.269	-0.5040	3.380	-0.4391	3.322	-0.2875	3.380	-0.4618	3.225	-0.6970
3.412	-0.2988	3.486	-0.1294	3.431	-0.1368	3.477	-0.1282	3.380	-0.4138
3.502	-0.1343	3.571	0.0567	3.534	-0.0238	3.556	0.0939	3.486	-0.1934
3.577	-0.0309	3.647	0.2026	3.598	0.0686	3.635	0.1294	3.564	-0.0594
3.697	-0.1406	3.713	0.3247	3.692	0.2121	3.692	0.2905	3.629	0.0854
3.782	0.2827	3.751	0.3939	3.791	0.3981	3.760	0.3660	3.692	0.2356
3.843	0.3857	3.808	0.4133	3.835	0.4156	3.827	0.4319	3.765	0.3315
3.902	0.4652	3.820	0.4680	3.868	0.4765	3.865	0.4765	3.868	0.3757
3.954	0.4111	-	-	3.912	0.5181	3.899	0.4544	3.905	0.4824
3.996	0.4492	-	-	3.948	0.5146	3.911	0.4794	3.930	0.4915
-	-	-	-	-	-	-	-	3.968	0.4492

TABLE 6: DATA OF LOG.ln $\frac{y}{yo-y}$ AND LOG.t USED IN LINEAR REGRESSION ANALYSES TO EVALUATE THE CONSTANTS β AND γ - CYCLIC ADSORPTION OPERATIONS INVOLVING TOLUENE

APPENDIX V

SUMMARY OF DATA USED IN EVALUATING THE LENGTH OF UNUSED ADSORBENT BED AT BREAKTHROUGH

Run No:	D (m)	G_s ($kg.s^{-1}.m^{-2}$)	t_B (s)	Yo $\frac{\text{wt. solvent}}{\text{wt. air}}$ ($kg.kg^{-1}$)	We $\frac{\text{wt. solvent}}{\text{wt. cloth}}$ ($kg.kg^{-1}$)	Ps ($kg.m^{-3}$)	$G_s t_B (Yo-Yo^*)$ (m)	L (m)
BP1	0.600	0.1664	2500	0.0215	0.753	65.205	0.1822	0.4178
BP4	0.600	0.1645	1600	0.0574	0.747	74.920	0.2774	0.3226
BP6	0.600	0.1662	300	0.1145	0.763	65.896	0.1136	0.4864
BP10	0.600	0.1648	1600	0.0573	0.830	66.000	0.2758	0.3242
BP12	0.600	0.0525	6500	0.0681	0.818	66.307	0.4285	0.1715
BP13	0.600	0.1027	1500	0.0697	0.811	66.454	0.1992	0.4008
BP14	0.600	0.1349	1500	0.0700	0.794	66.190	0.3161	0.2839
BT1	0.600	0.1662	5200	0.0115	0.319	67.865	0.4591	0.1409
BT5	0.600	0.1649	2500	0.0306	0.371	65.514	0.5190	0.0810
BT7	0.600	0.1664	200	0.0611	0.388	64.603	0.0828	0.5172
BT8	0.600	0.0526	7800	0.0363	0.360	70.451	0.5872	0.0128
BT9	0.600	0.1011	400	0.0378	0.370	65.000	0.0636	0.5364
BT10	0.600	0.1338	1250	0.0377	0.351	68.982	0.2604	0.3396

TABLE 1: DATA USED IN EVALUATING THE LENGTH OF UNUSED ADSORBENT BED AT BREAKTHROUGH FOR SELECTED SINGLE ADSORPTION OPERATIONS

Run No:	D (m)	G_s (kg.s^{-1}.m^{-2})	t_B (s)	Yo $\frac{\text{wt. solvent}}{\text{wt. air}}$ (kg.kg^{-1})	Yo* $\frac{\text{wt. solvent}}{\text{wt. air}}$ (kg.kg^{-1})	We $\frac{\text{wt. solvent}}{\text{wt. cloth}}$ (kg.kg^{-1})	Wo $\frac{\text{wt. solvent}}{\text{wt. cloth}}$ (kg.kg^{-1})	Ps (kg.m^{-3})	$\frac{G_s t_B (Yo-Yo^*)}{Ps (We-Wo)}$ (m)	L (m)
CBP1	0.600	0.1665	2000	0.0859	0.0	0.816	0.0	66.422	0.5280	0.0720
CBP2	0.600	0.1665	650	0.0859	0.0	0.813	0.248	83.953	0.1968	0.4032
CBP3	0.600	0.1665	650	0.0859	0.0	0.776	0.242	82.483	0.2110	0.3890
CBP4	0.600	0.1665	500	0.0859	0.0	0.784	0.322	87.832	0.1762	0.4238
CBT1	0.600	0.1665	1000	0.0459	0.0	0.357	0.0	67.847	0.3155	0.2845
CBT2	0.600	0.1665	1000	0.0459	0.0	0.365	0.196	81.146	0.5573	0.1527
CBT3	0.600	0.1665	1000	0.0459	0.0	0.348	0.102	74.754	0.4156	0.1844
CBT4	0.600	0.1665	1000	0.0459	0.0014	0.350	0.243	84.306	0.8208	-0.2208

TABLE 2: DATA USED IN EVALUATING THE LENGTH OF UNUSED ADSORBENT BED AT BREAKTHROUGH FOR THE CYCLIC ADSORPTION OPERATIONS

APPENDIX VI

TABLES OF THE ADSORPTION RESULTS FROM OPERATIONS CARRIED OUT ON THE CONTINUOUS SYSTEM

SAMPLE POINT 1			SAMPLE POINT 2			SAMPLE POINT 3			SAMPLE POINT 4			SAMPLE POINT 5			SAMPLE POINT 6			OUTLET SAMPLE POINT		
Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	900	0.0190	0.386
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1200	0.0143	0.290
1260	0.0436	0.886	1320	0.0336	0.682	1380	0.0298	0.605	1440	0.0249	0.507	1500	0.0273	0.555	1560	0.0255	0.518	1860	0.0158	0.321
																		2460	0.0190	0.386
2520	0.0406	0.826	-	-	-	2640	0.0381	0.775	-	-	-	2700	0.0328	0.667	-	-	-	2760	0.0204	0.414
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3360	0.0273	0.555
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3840	0.0295	0.600
-	-	-	-	-	-	-	-	-	4020	0.0416	0.845	-	-	-	-	-	-	5220	0.0285	0.580
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5820	0.0392	0.797
6180	0.0438	0.891	-	-	-	6000	0.0410	0.833	-	-	-	6360	0.0383	0.778	-	-	-	7200	0.0459	0.933
-	-	-	7820	0.0452	0.918	-	-	-	-	-	-	7500	0.0444	0.903	-	-	-	8520	0.0338	0.686

TABLE 1: ADSORPTION RESULTS FOR EXPERIMENTAL RUN CP1

SAMPLE POINT 1			SAMPLE POINT 2			SAMPLE POINT 3			SAMPLE POINT 4			SAMPLE POINT 5			SAMPLE POINT 6			OUTLET SAMPLE POINT		
Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration	Fraction of inlet concentration
RUN CP2A																				
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	660	0.0238	0.483
780	0.0398	0.809	-	-	-	-	-	-	720	0.0356	0.724	-	-	-	-	-	-	1620	0.0217	0.442
-	-	-	-	-	-	1680	0.0412	0.838	-	-	-	-	-	-	-	-	-	2400	0.0254	0.516
2580	0.0435	0.884	2700	0.0424	0.862	2760	0.0373	0.758	2940	0.0391	0.795	3060	0.0342	0.696	3240	0.0328	0.666	3360	0.0220	0.447
RUN CP2B																				
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	360	0.0341	0.554
-	-	-	600	0.0480	0.780	-	-	-	720	0.0412	0.670	-	-	-	1020	0.0406	0.660	1380	0.0446	0.725
1500	0.0526	0.855	1620	0.0523	0.851	1860	0.0436	0.705	2040	0.0490	0.796	2160	0.0434	0.706	2340	0.0421	0.684	2520	0.0356	0.579
-	-	-	-	-	-	-	-	-	3180	0.0559	0.909	-	-	-	-	-	-	3300	0.0371	0.603
-	-	-	3480	0.0536	0.872	-	-	-	-	-	-	-	-	-	-	-	-	4800	0.0501	0.814
4980	0.0549	0.892	5460	0.0540	0.878	5880	0.0518	0.843	-	-	-	5640	0.0476	0.774	5220	0.0473	0.769	7020	0.0464	0.755
-	-	-	-	-	-	-	-	-	7140	0.0512	0.832	-	-	-	-	-	-	9480	0.0630	1.024
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	11040	0.0661	1.075
RUN CP2C																				
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	480	0.0468	0.761
-	-	-	1200	0.0582	0.947	660	0.0502	0.816	-	-	-	1560	0.0447	0.727	-	-	-	1020	0.0466	0.757
-	-	-	2820	0.0557	0.906	3240	0.0518	0.842	-	-	-	2520	0.0469	0.763	-	-	-	2220	0.0442	0.719

TABLE 2: ADSORPTION RESULTS FOR EXPERIMENTAL RUNS CP2A, CP2B AND CP2C

	SAMPLE POINT 1			SAMPLE POINT 2			SAMPLE POINT 3			SAMPLE POINT 4			SAMPLE POINT 5			SAMPLE POINT 6			OUTLET SAMPLE POINT		
	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration
E	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	480	0.0157	0.680
M	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-			
E	-	-	-	-	-	-	750	0.0148	0.642	-	-	-	-	-	-	-	-	-	1200	0.0148	0.640
M	-	-	-	-	-	-	810	0.0132	0.573	-	-	-	-	-	-	-	-	-			
E	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1680	0.0150	0.649
M	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-			
E	1860	0.0169	0.731	-	-	-	-	-	-	-	-	-	-	-	-	3060	0.0160	0.692	3120	0.0160	0.692
M	1980	0.0151	0.656	-	-	-	-	-	-	-	-	-	-	-	-	2940	0.0105	0.454			
E	-	-	-	-	-	-	3360	0.0169	0.733	-	-	-	-	-	-	-	-	-	3720	0.0157	0.681
M	-	-	-	-	-	-	3540	0.0167	0.721	-	-	-	-	-	-	-	-	-			
E	-	-	-	4980	0.0128	0.943	-	-	-	-	-	-	4230	0.0159	0.689	-	-	-	5700	0.0116	0.503
M	-	-	-	5220	0.0183	0.792	-	-	-	-	-	-	4290	0.0157	0.680	-	-	-			
E	-	-	-	-	-	-	-	-	-	5820	0.0156	0.676	-	-	-	-	-	-	7140	0.0134	0.582
M	-	-	-	-	-	-	-	-	-	5880	0.0074	0.320	-	-	-	-	-	-			
E	-	-	-	8460	0.0184	0.795	-	-	-	-	-	-	9000	0.0184	0.795	-	-	-	8160	0.0179	0.777
M	-	-	-	8700	0.0160	0.691	-	-	-	-	-	-	9120	0.0175	0.757	-	-	-			

TABLE 3: ADSORPTION RESULTS FOR EXPERIMENTAL RUN CT1A

	SAMPLE POINT 1			SAMPLE POINT 2			SAMPLE POINT 3			SAMPLE POINT 4			SAMPLE POINT 5			SAMPLE POINT 6			OUTLET SAMPLE POINT		
	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration	Time (secs)	Volumetric concentration (vol %)	Fraction of inlet concentration
E	-	-	-	-	-	-	-	-	-	1860	0.0182	0.790	-	-	-	2460	0.0192	0.831	1020	0.0146	0.633
M	-	-	-	-	-	-	-	-	-	1560	0.0047	0.205	-	-	-	3000	0.0156	0.677	4440	0.0139	0.603
E	7590	0.0183	0.794	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	6600	0.0156	0.677
M	7980	0.0101	0.438	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	9000	0.0157	0.681
E	-	-	-	-	-	-	10350	0.0143	0.621	-	-	-	9480	0.0149	0.647	-	-	-	10920	0.0156	0.677
M	-	-	-	-	-	-	10620	0.0171	0.741	-	-	-	10080	0.00158	0.683	-	-	-	11460	0.0155	0.672
E	-	-	-	-	-	-	-	-	-	13380	0.0230	0.995	-	-	-	-	-	-	14940	0.0151	0.655
M	-	-	-	-	-	-	-	-	-	13620	0.0159	0.688	-	-	-	-	-	-	17520	0.0162	0.702
E	-	-	-	15600	0.0156	0.675	17820	0.0188	0.813	15240	0.0173	0.751	-	-	-	17100	0.0154	0.666	18720	0.0188	0.816
M	-	-	-	15720	0.0161	0.697	18060	0.0189	0.820	15480	0.0131	0.568	-	-	-	17340	0.0150	0.648	19680	0.0199	0.861
M	19020	0.0195	0.844	20820	0.0158	0.682	-	-	-	21180	0.0166	0.719	-	-	-	22140	0.0154	0.668	22440	0.0169	0.732
E	-	-	-	-	-	-	-	-	-	21720	0.0195	0.846	-	-	-	-	-	-	-	-	-

TABLE 4: ADSORPTION RESULTS FOR EXPERIMENTAL RUN CT1B

APPENDIX VII

HEAT OF WETTING AND SATURATION VAPOUR UPTAKES WITH RESPECT TO CHARCOAL CLOTH WHICH HAS UNDERGONE A VARYING NUMBER OF REGENERATIONS

	Heats of wetting J g^{-1}				Benzene SVU				Carbon tetrachloride SVU			
	Benzene		Silicone 2 cP		Oven dried		P_2O_5 dried		Oven dried		P_2O_5 dried	
Sample No.	Oven dried	P_2O_5 dried	Oven dried	P_2O_5 dried	% wt/wt	cc g^{-1}	% wt/wt	cc g^{-1}	% wt/wt	cc g^{-1}	% wt/wt	cc g^{-1}
1	130.7	29.3	117.9	45.5	60.4	0.689	31.1	0.355	116.0	0.729	72.6	0.456
2	127.2	30.8	111.5	34.7	59.5	0.678	31.9	0.364	114.5	0.719	72.0	0.452
3	127.9	30.1	111.5	45.8	65.5	0.747	31.0	0.353	116.9	0.734	69.7	0.438
Average	128.5	30.1	113.6	42.0		0.705		0.357		0.727		0.449
4	43.9	36.0	48.2	41.9	23.9	0.273	18.8	0.214	65.5	0.411	60.3	0.379
5	33.1	28.8	33.4	38.0	16.7	0.190	25.2	0.287	59.5	0.374	65.2	0.410
6	52.4	34.6	43.4	49.7	28.7	0.327	25.8	0.294	68.2	0.428	63.3	0.398
Average	43.1	33.1	41.7	43.2		0.263		0.265		0.404		0.396
7	38.0	18.9	30.6	44.4	20.1	0.229	19.4	0.221	56.6	0.356	59.2	0.372
8	40.4	30.0	35.5	42.8	14.3	0.163	15.8	0.180	51.4	0.323	54.4	0.342
9	40.6	25.1	36.4	41.8	11.4	0.130	13.9	0.158	48.7	0.306	47.8	0.300
Average	39.7	24.7	34.2	43.0		0.225		0.186		0.328		0.338
10	51.1	40.3	50.1	43.7	24.9	0.283	14.2	0.162	67.6	0.425	45.1	0.283
11	53.4	32.1	49.5	42.6	24.4	0.278	13.5	0.154	63.2	0.397	45.5	0.286
12	33.6	31.9	40.5	51.6	26.2	0.299	22.8	0.260	58.7	0.369	53.6	0.337
Average	46.0	34.8	46.7	46.0		0.287		0.192		0.397		0.302
13	94.5	43.7	80.0	47.2	55.4	0.632	31.4	0.358	108.2	0.680	80.4	0.505
14	82.3	25.7	81.5	50.1	53.9	0.615	36.3	0.414	102.1	0.641	63.7	0.400
15	101.0	32.9	85.5	58.9	54.4	0.620	26.9	0.307	106.8	0.671	74.8	0.470
Average	92.6	34.1	82.3	52.1		0.622		0.360		0.664		0.458

TABLE 1: HEATS OF WETTING AND SATURATION VAPOUR UPTAKES

APPENDIX VIII

DEMAND FOR AND DISTRIBUTION OF ACTIVATED CARBON IN THE UNITED STATES

UNITED STATES' DEMAND FOR ACTIVATED CARBON (millions of kg)

Applications	1976		Average Annual Growth Rate %
LIQUID PHASE			
Sugar Decolourizing	23.1		1.50
Cane		4.2	
Beet		1.8	
Corn Syrups		17.3	
Drinking Water	17.3		5.5
Taste and Odour Control		17.3	
Organic Compounds		-	
Waste Water	10.4		15.5
Municipal		4.5	
Industrial		5.9	
Fats, Oils, Foods		4.5	2.0
Pharmaceuticals		3.2	
Dry Cleaning		3.2	
Electroplating		1.4	
Reclaimed Rubber		0.4	
Miscellaneous		7.7	
Total - Liquid Phase	71.2		5.0
GAS PHASE			
Automotive		4.3	5.5
Cigarettes		2.3	
Solvent Vapour Recovery		1.4	
Air Purification		1.1	
Catalysts		1.0	
Gas Masks		0.5	
Nuclear		0.3	
Miscellaneous		1.4	
Total - Gas Phase	12.3		5.5
GRAND TOTAL	83.5		5.0

TABLE 1: DEMAND FOR AND DISTRIBUTION OF ACTIVATED CHARCOAL IN THE UNITED STATES