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***Manufacturing of Activated
Carbon Filter to Remove
Chromium Oxide and Tea Dye
from Water***

A Thesis

*Submitted to the College of Engineering of Babylon
University in Partial Fulfillment of the Requirements
for the award of Degree of Master of Science in Materials
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(Ceramic Materials)*

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

أَفَإَيْتُرُ الْمَاءَ الَّذِي تَسْجُونَ  أَنْتُمْ
أَنْزَلْنَاهُ مِنَ الْمَزْنِ أَمْ نَحْنُ
الْمُنزِلُونَ 
فَلَوْلَا تَشْكُرُونَ 

سُورَةُ الرَّحْمَنِ الرَّحِيمِ

سورة الواقعة الآية (68-70)

الخلاصة

تتناول هذه الدراسة إنتاج مرشحات كربونية على هيئة كتل صلبة (SBACF) من الكربون المنشط ومن المادة الأولية المحلية (البولي اثيلين عالي الكثافة), ودراسة أمكانية إزالة صبغتي الشاي واوكسيد الكروم السداسي من الماء بواسطة هذا المرشح بالإضافة إلى دراسة خواص المرشح الفيزيائية (المسامية الظاهرية, الكثافة الكتلية, النفاذية وقياس اكبر قطر للمسامة) والميكانيكية (مقاومة الانضغاط والصلادة).

تم استخدام ثلاثة حجوم حبيبية للكربون المنشط وهي (75, 90, 150) مايكروميتر حيث تم خلط كل حجم حبيبي مع ثلاث نسب وزنيه مختلفة للمادة الرابطة وهي (10, 20, 30)% من وزن النموذج, بعد ذلك تم تسخين الخليط في الفرن بدرجة (200 م°) وذلك لغرض جعل المادة الرابطة لزجة لتعمل على ربط حبيبات الكربون ثم تم كبس النماذج تحت ضغوط مختلفة (10, 30, 20) طن وذلك لمعرفة تأثير كل من (الحجم الحبيبي للكربون, نسبة المادة الرابطة وضغط الكبس) على كفاءة وخواص المرشح الكربوني الأخرى.

بعد ذلك تم إجراء عدة فحوصات فيزيائية وميكانيكية وكيمائية حيث تم الحصول عمليا على تأثير المتغيرات اعلاة على خواص المرشح الكربوني فوجد إن كل من المسامية والنفاذية واكبر قطر للفجوة تزداد بزيادة الحجم الحبيبي للكربون وتقل بزيادة نسبة المادة الرابطة وضغط الكبس المستخدم, أما الكثافة فأنها تزداد بزيادة كل من نسبة الرابطة وضغط الكبس وتقل بزيادة الحجم الحبيبي للكربون.

في حين إن مقاومة الانضغاط وصلادة النماذج تزداد بزيادة نسبة الرابطة والضغط وتقل بزيادة الحجم الحبيبي للكربون, ولكن كفاءة الفلتر على امتزاز صبغة الشاي واوكسيد الكروم السداسي تقل بزيادة الحجم الحبيبي للكربون ونسبة المادة الرابطة وتزداد بزيادة ضغط الكبس المستخدم.

تصل كفاءة المرشح في إزالة صبغة الشاي من الماء إلى 99.9% وبتركيز وصل إلى 0.0824 جزء في المليون أما في إزالة اوكسيد الكروم السداسي فتصل إلى 99.8% وبتركيز 0.1738 جزء في المليون.



Abstract

This work is conducted to study the ability for producing solid block activated carbon filter from activated carbon (AC) and local raw material represented by high density polyethylene (HDPE) and study the ability of removing contaminants (tea dye and CrO_3) from water using solid activated carbon filter by the adsorption mechanism.

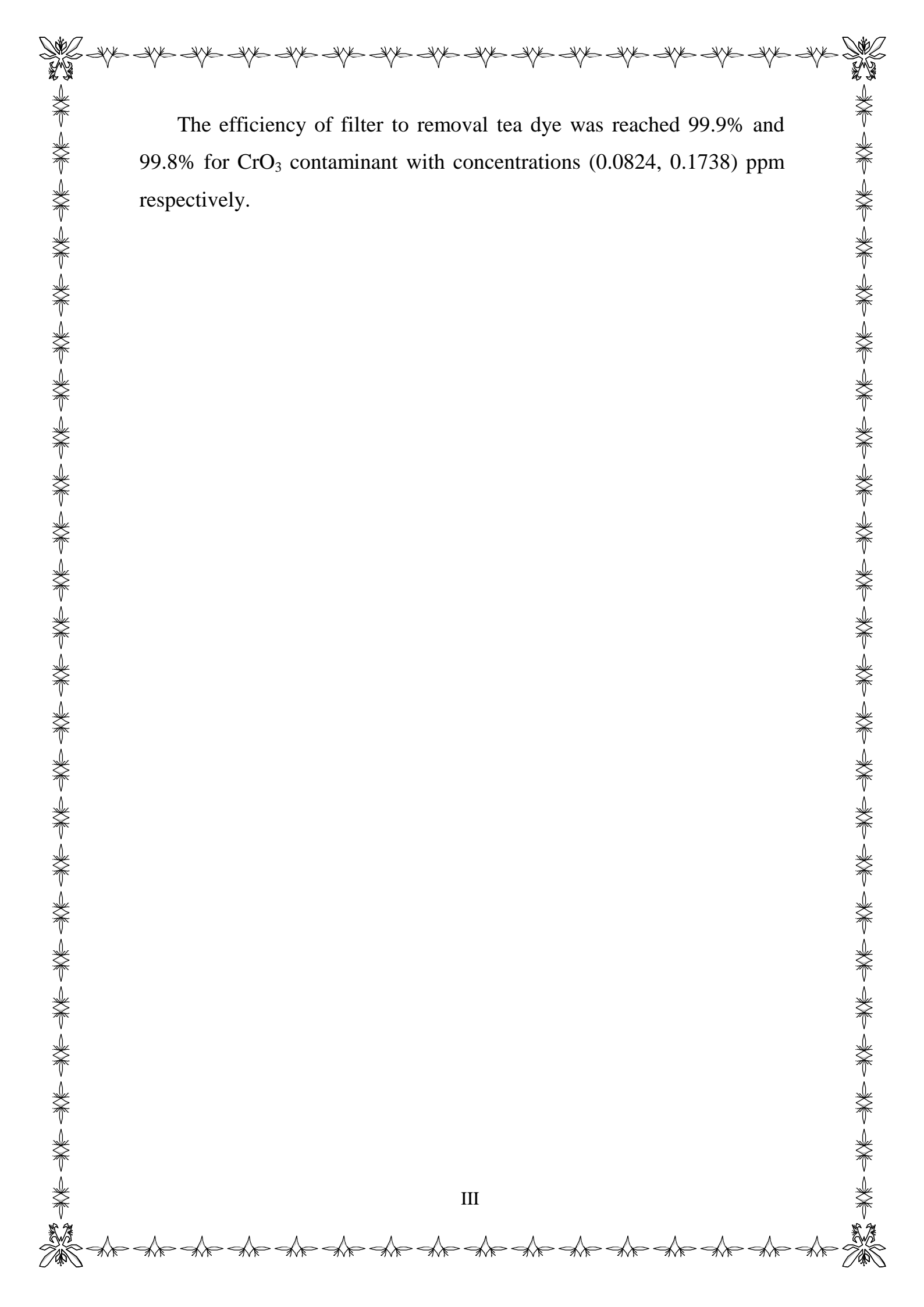
Three particle sizes of carbon powder were used in this study (75, 90 and $150\mu\text{m}$). Each single particle size mixed with three different percents of HDPE which are (10, 20, 30) % from the sample weight. After that the mixture was heated in the oven at $200\text{ }^\circ\text{C}$ to make the binder material become tacky to sufficiently wet the carbon particles. The powder was pressed in the mold under three different load values (10, 20 and 30) tons to specify the effect of these factors (carbon particle size, binder percent and compaction pressure) on carbon filter properties.

Physical, mechanical and chemical tests have been done on filter samples and experimentally tested the effect of factors mentioned above on the filter samples properties.

Porosity, permeability and max. pore diameter were increased with increasing carbon particle size and were decreased with increasing binder percent and compaction pressure, while density of the body increased with increasing binder percent and compaction pressure and was decreased with increasing carbon particle size.

Both compressive strength and hardness of filter samples were increased with increasing binder percent and compaction pressure, and were decreased with increasing carbon particle size.

The ability of filter to remove tea dye and CrO_3 from water by adsorption process was increased with decreasing each of carbon particle size, binder percent and compaction pressure.



The efficiency of filter to removal tea dye was reached 99.9% and 99.8% for CrO₃ contaminant with concentrations (0.0824, 0.1738) ppm respectively.

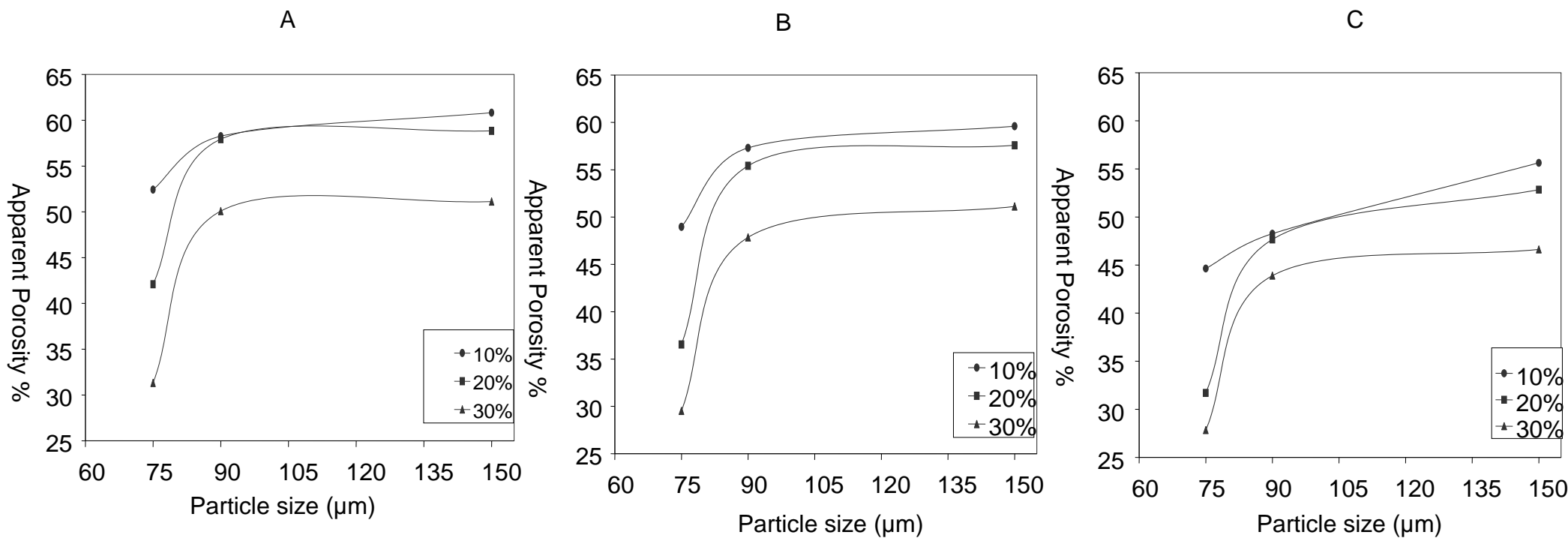


Fig. 4-1: The effect of carbon particle size on apparent porosity.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

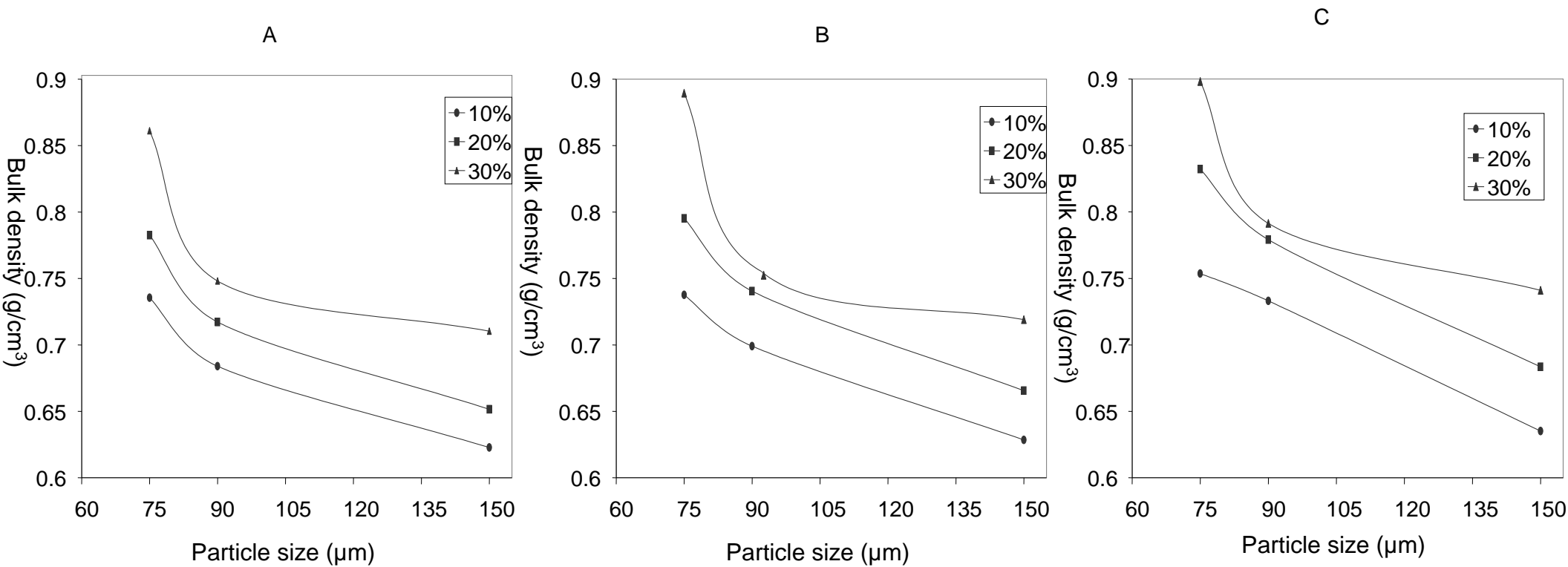


Fig. 4-2: The effect of carbon particle size on the bulk density.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

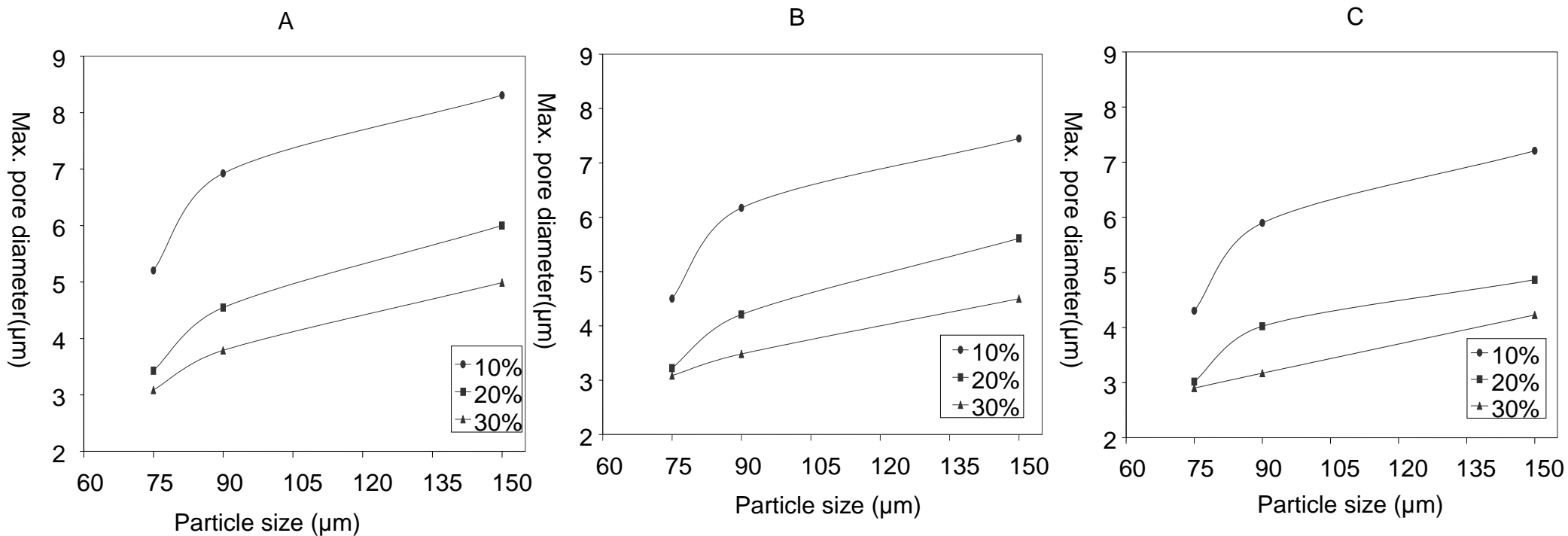


Fig. 4-3: The effect of carbon particle size on the experimental Max. Pore Diameter.

A) - at (10 ton ≡ 122 MPa compaction pressure), B)-at (20 ton ≡ 244 MPa compaction pressure), C) – at (30 ton ≡ 365 MPa compaction pressure)

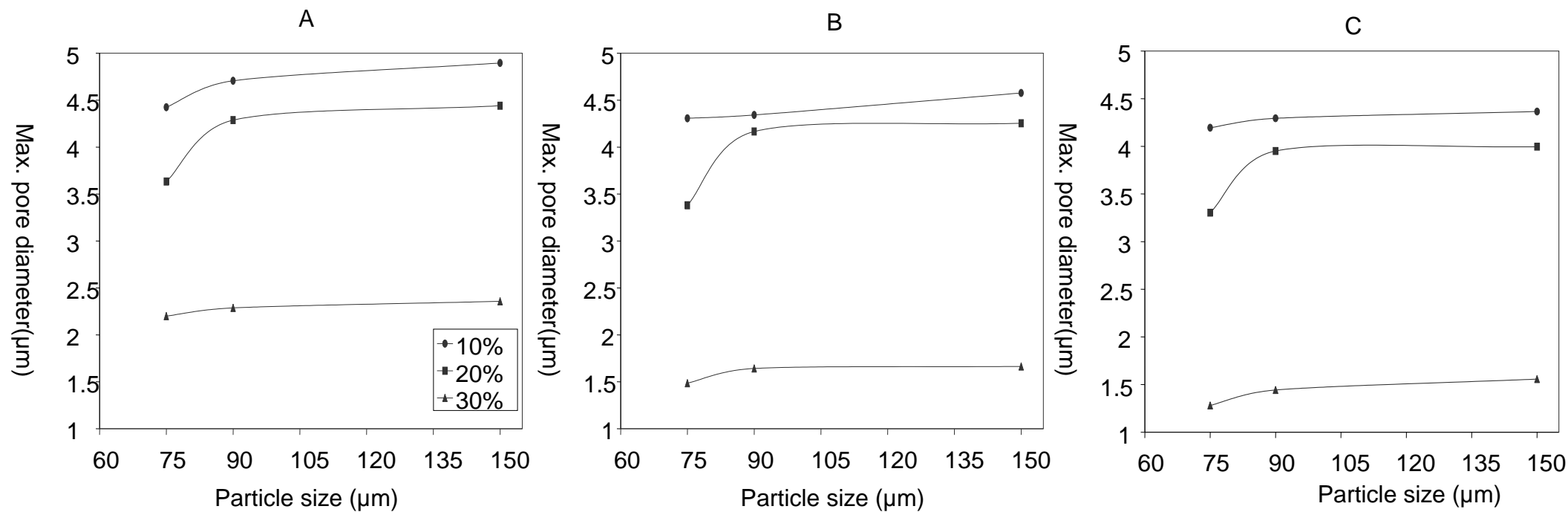


Fig. 4-4: The effect of carbon particle size on the theoretical Max. Pore Diameter.

A) - at (10 ton ≡ 122 MPa compaction pressure), B)-at (20 ton ≡ 244 MPa compaction pressure), C) – at (30 ton ≡ 365 MPa compaction pressure)

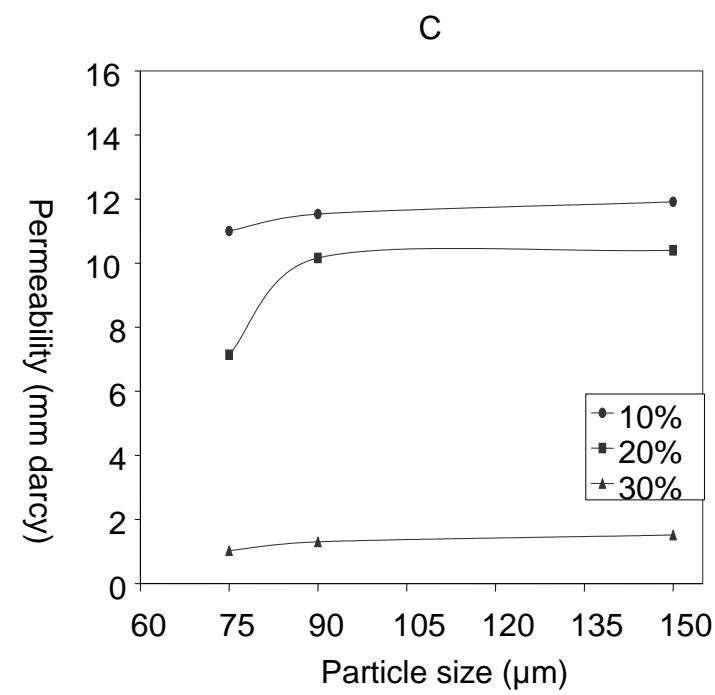
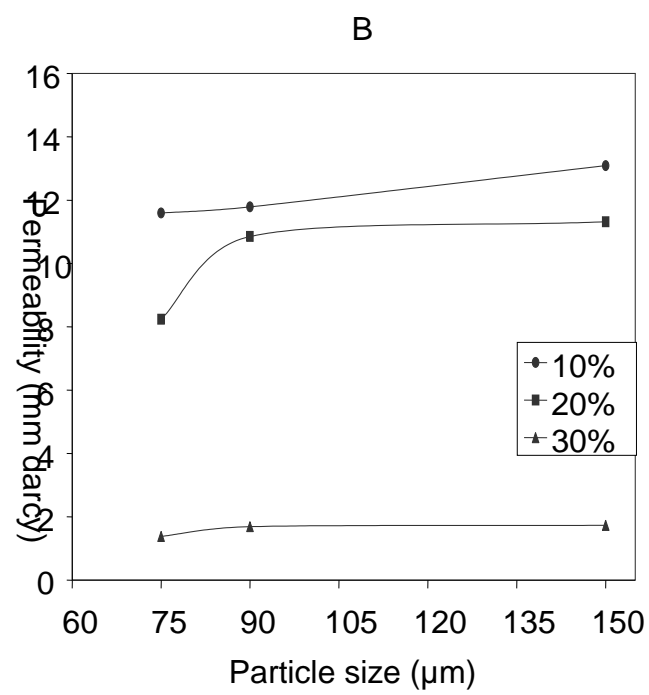
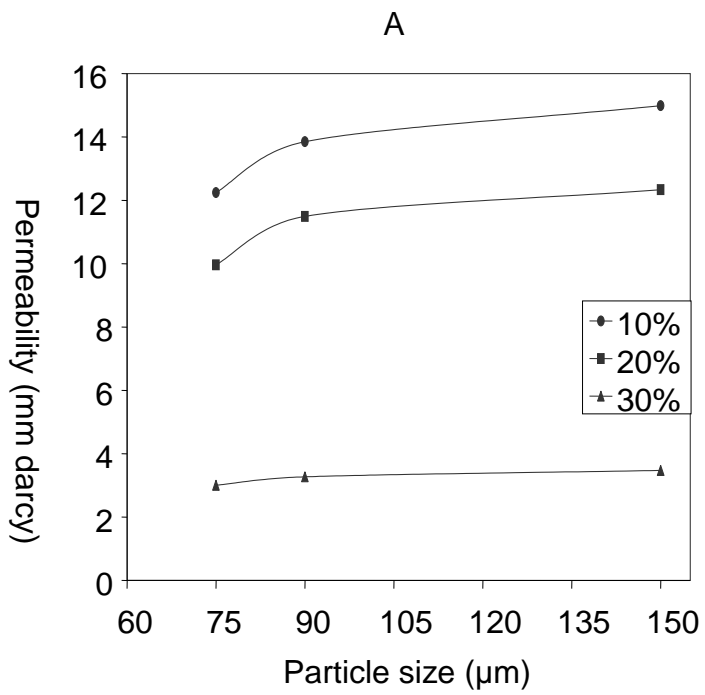


Fig. 4-5: The effect of carbon particle size on the permeability.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

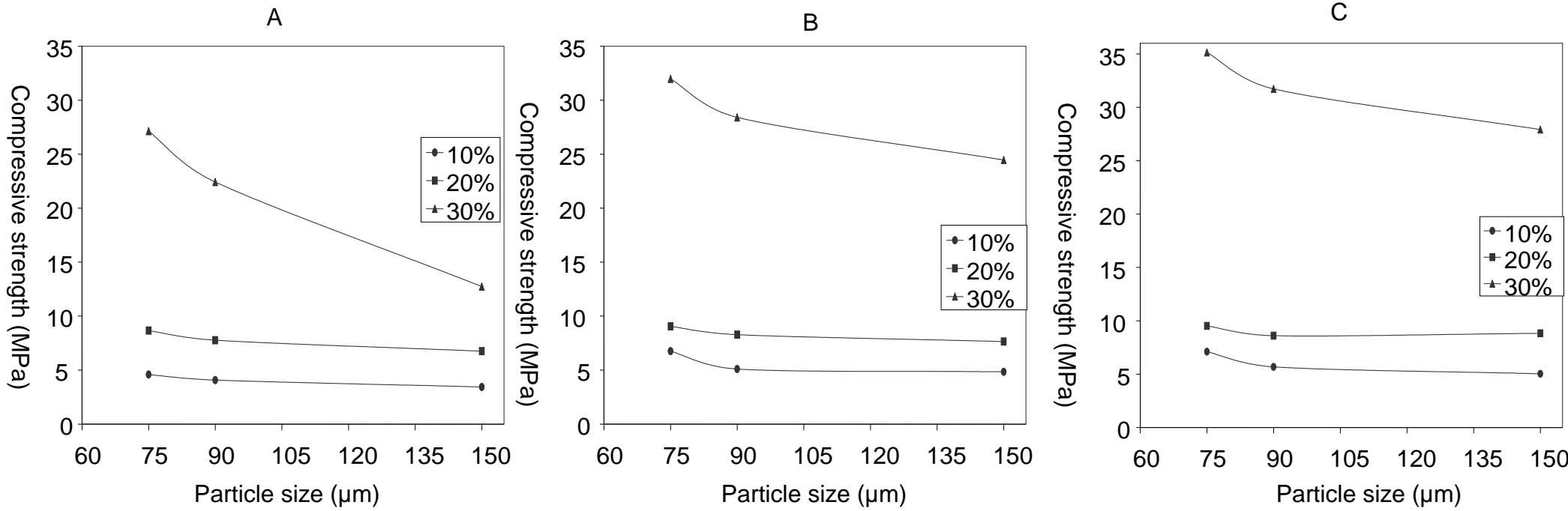


Fig. 4-6: The effect of carbon particle size on the compressive strength.

A) - at (0.977 ton≡ 122 MPa compaction pressure), B)-at (1.954 ton≡ 244 MPa compaction pressure), C) – at (2.923 ton≡ 365 MPa compaction pressure)

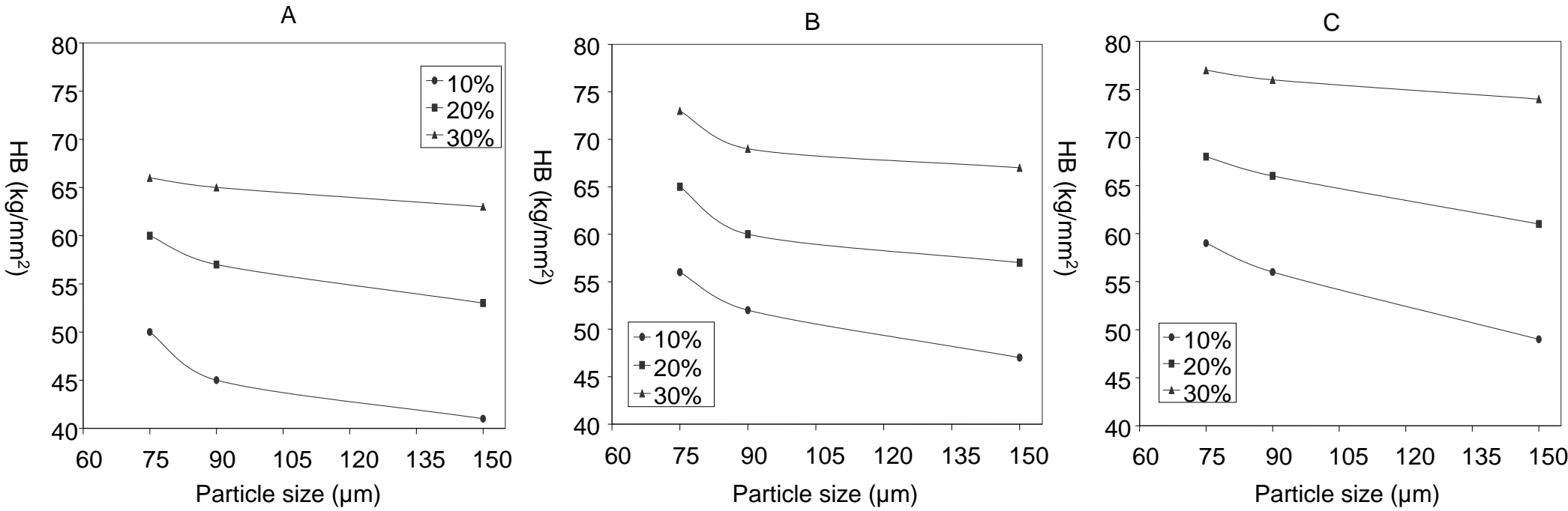


Fig. 4-7: The effect of carbon particle size on the hardness.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

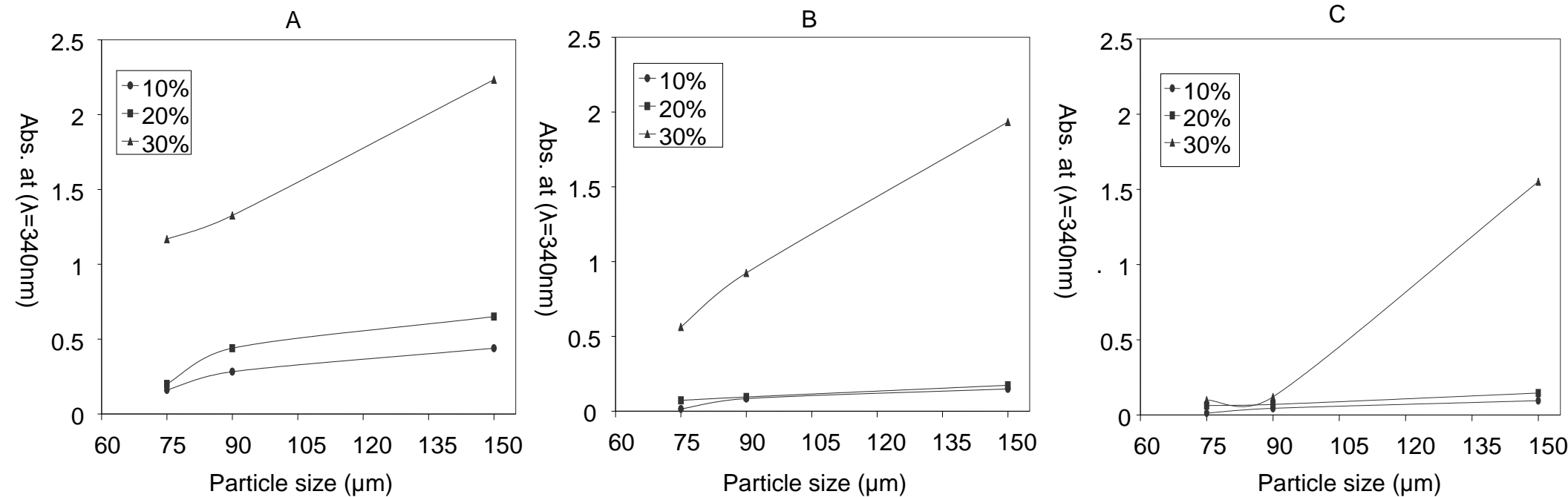


Fig. 4-8: The effect of carbon particle size on the absorption process for tea dye.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

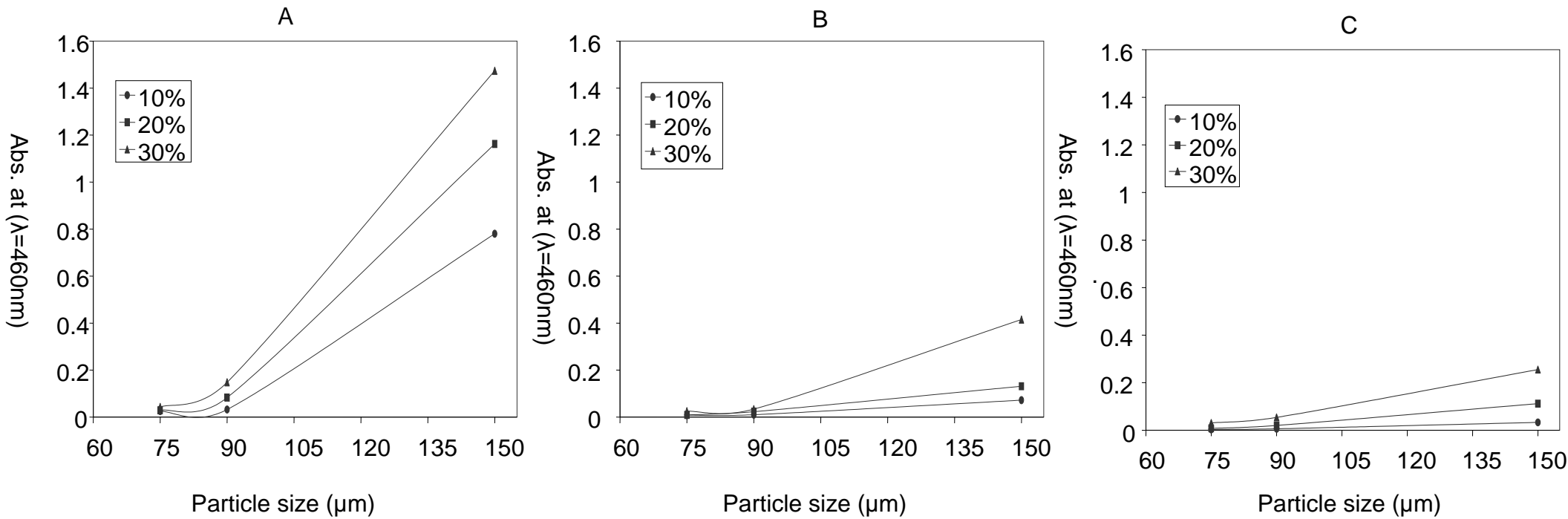


Fig. 4-9: The effect of carbon particle size on the absorption process for CrO₃.

A) - at (10 ton≡ 122 MPa compaction pressure), B)-at (20 ton≡ 244 MPa compaction pressure), C) – at (30 ton≡ 365 MPa compaction pressure)

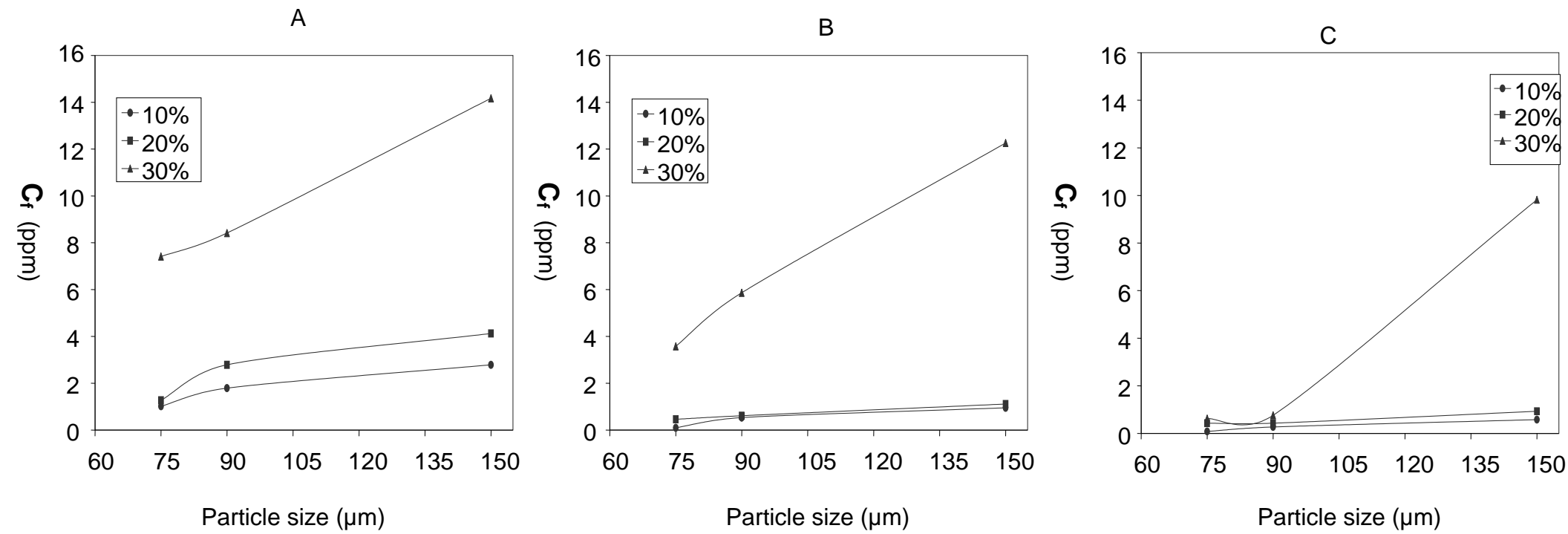


Fig. 4-10: The effect of carbon particle size on the tea dye final concentration (C_f).

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

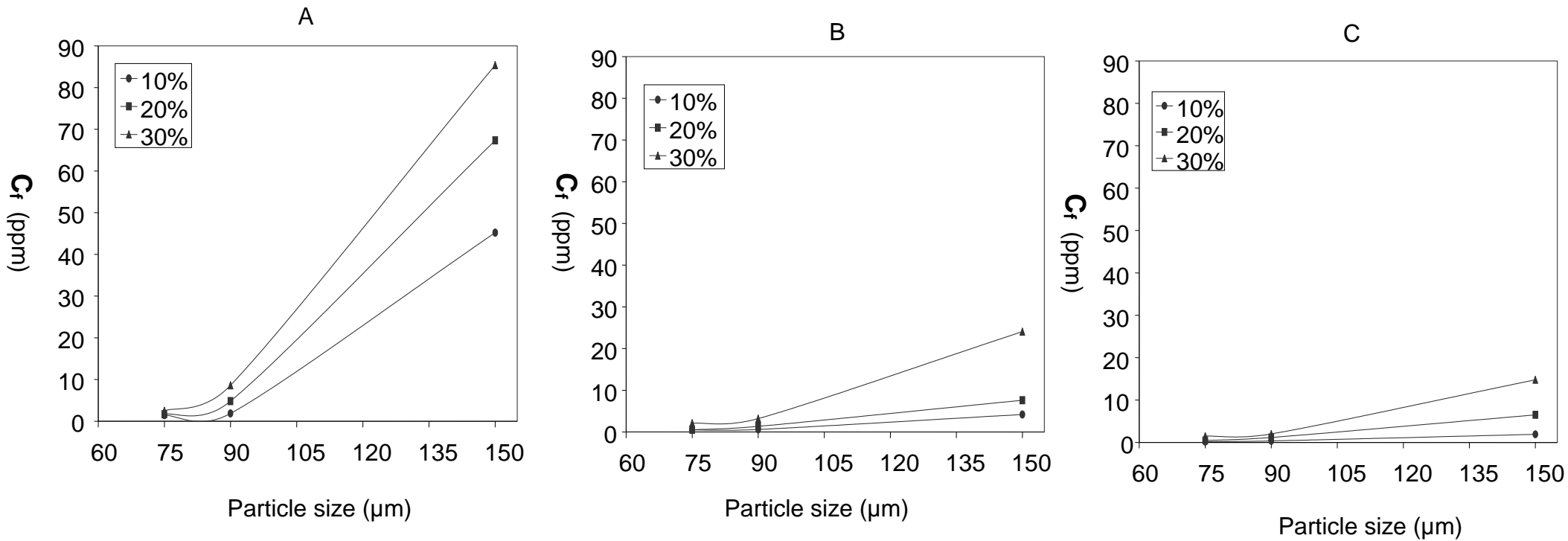


Fig. 4-11: The effect of carbon particle size on the CrO_3 final concentration (C_f).

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

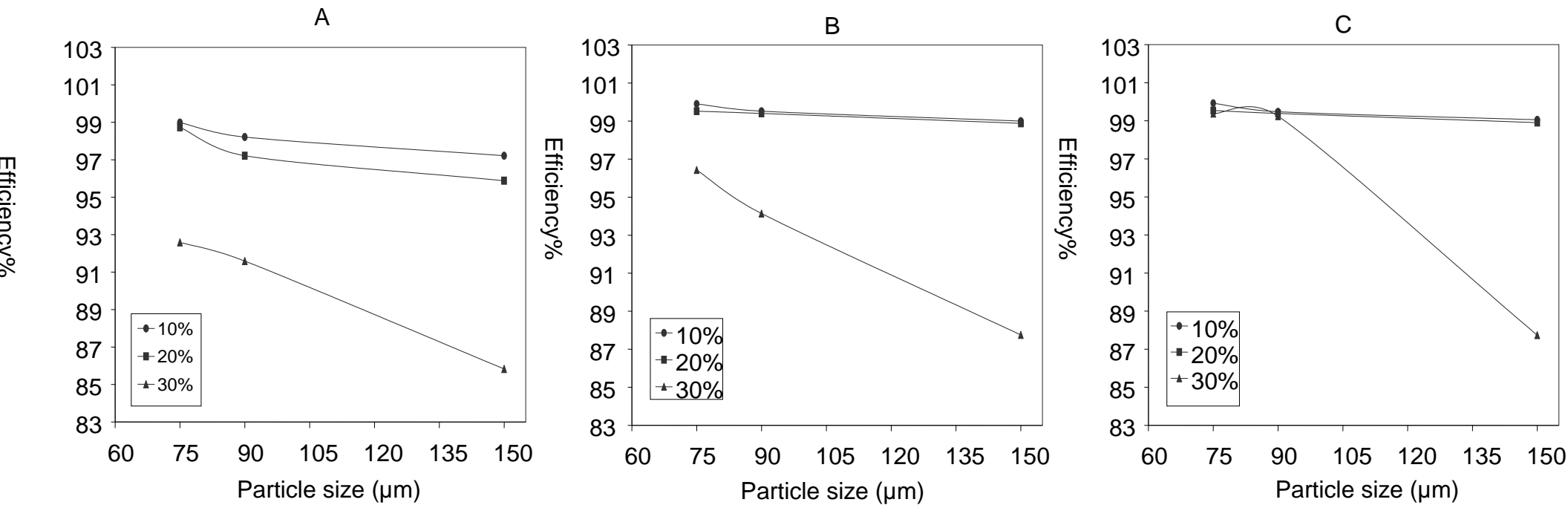


Fig. 4-12: The effect of carbon particle size on the tea dye removal efficiency.

A) - at (10 ton ≡ 122 MPa compaction pressure), B)-at (20 ton ≡ 244 MPa compaction pressure), C) – at (30 ton ≡ 365 MPa compaction pressure)

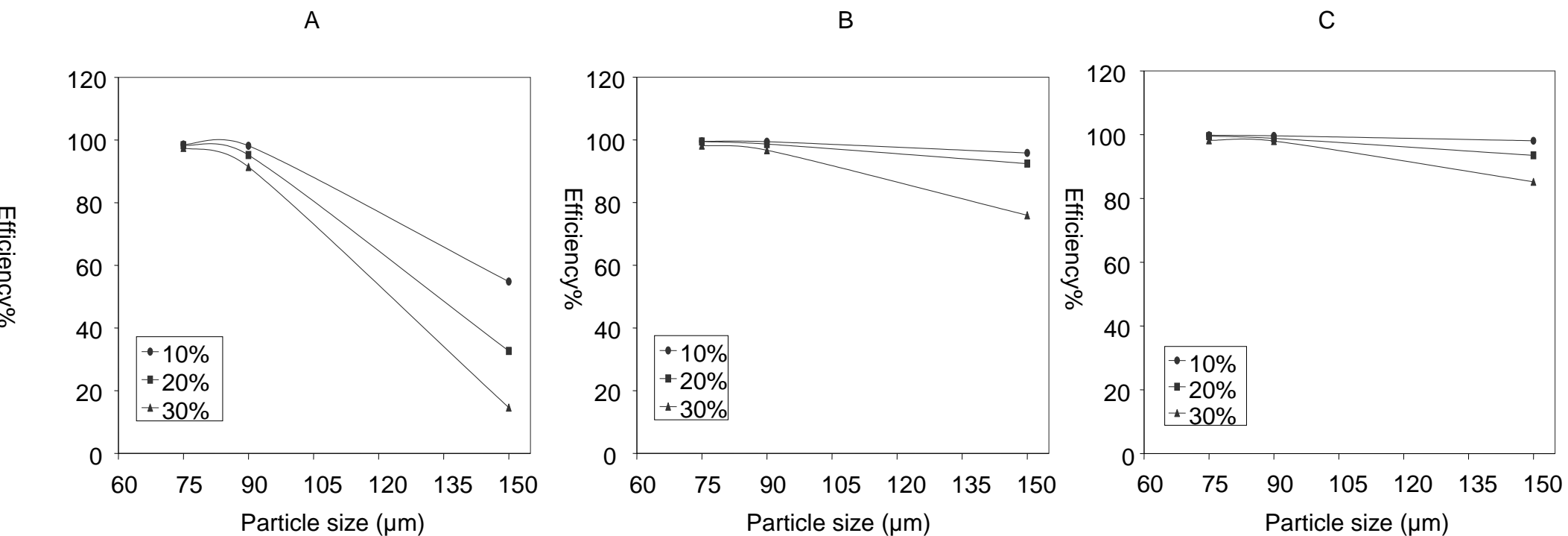


Fig. 4-13: The effect of carbon particle size on the CrO₃ removal efficiency.

A) - at (10 ton \equiv 122 MPa compaction pressure), B)-at (20 ton \equiv 244 MPa compaction pressure), C) – at (30 ton \equiv 365 MPa compaction pressure)

CERTIFICATE

We certify that we have read this thesis, titled “**Manufacturing of Activated Carbon Filter to Remove Chromium Oxide and Tea Dye from Water**”, and as examining committee examined the student “*Kutaiba H. Mohammed AL-Marzoki*” in its contents and in what is connected with it, and that in our opinion it meets the standard of a thesis for the degree of Master of Science in Materials Engineering.

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I dedicate this thesis to:

My parents,

My fiancé,

&

My brothers

For their encouragement, support,

and love.

Kutaiba

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First of all I thank *Allah* for every thing.

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Kutaiba

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NOMENCLATURE

<i>Symbol</i>	<i>Description</i>	<i>Unit</i>
V	Exterior volume of sample	cm³
M	Saturated weight of sample	g
S	Suspended weight of sample	g
D	Dry weight of sample	g
P	Apparent porosity	%
A	Water absorption	%
B	Bulk density	g/ cm³
T	True density	g/ cm³
Q	Volumetric Flow	cm³/sec.
K	Permeability	Darcy's= (cm².cP)/(sec.atm)
A_r	cross-section area	cm²
μ	Viscosity	cP
P_r	Pressure	Atm. or mmHg
x	Length	mm
D_{max.}	Maximum pore diameter	μm
γ	Surface tension of test liquid	dynes/cm
σ_c	Compressive strength	MPa
F	Load applied until fracture	N
Abs.	Absorbency	-
α	The extinction coefficient	-
C	Dye concentration	ppm
L	The path length of the cell	mm
σ_f	Fracture strength	MPa
k	Constant depending on the material properties	-
d	Grain size diameter	μm
HB	Brinell hardness	kg/mm²
ε	Filter efficiency	%
C_o	Initial dye concentration	ppm
C_f	Final dye concentration	ppm

ABBREVIATIONS

<i>Symbol</i>	<i>Description</i>
<i>SBACF</i>	<i>Solid Block Activated Carbon Filter</i>
<i>AC</i>	<i>Activated Carbon</i>
<i>HDPE</i>	<i>High-Density Polyethylene</i>
<i>F.C.C</i>	<i>Face Centered Cubic</i>
<i>GAC</i>	<i>Granular Activated Carbon</i>
<i>PAC</i>	<i>Powdered Activated Carbon</i>
<i>LDPE</i>	<i>Low Density Polyethylene</i>
<i>LLDPE</i>	<i>Linear Low-Density Polyethylene</i>
<i>VLDPE</i>	<i>Very-Low-Density Polyethylene</i>
<i>MDPE</i>	<i>Medium-Density Polyethylene</i>
<i>UHMWPE</i>	<i>Ultra-High-Molecular-Weight Polyethylene</i>
<i>EPA</i>	<i>United States Environmental Protection Agency</i>
<i>ppm</i>	<i>Part Per Million</i>
<i>WQA</i>	<i>Water Quality Association</i>
<i>CB</i>	<i>Carbon Block</i>

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Chapter One

INTRODUCTION

1-1 Introduction

Water is the most important resource. Without water life is not possible. From a chemical point of view, water, H₂O, is a pure compound, but in reality, you seldom drink, see, touch or use pure water. Water from various sources contains dissolved gases, minerals, organic and inorganic substances. ⁽¹⁾

In its pure state, water is one of the most aggressive solvents known. Called the “universal solvent,” water, to a certain degree, will dissolve and destruct virtually everything to which it is exposed. Pure water has a very high energy state and, like everything else in nature, seems to achieve energy equilibrium with its surroundings. It will dissolve the quantity of material available until the solution reaches saturation, the point at which no higher level of solids can be dissolved. Contaminants found in water include atmospheric gases, minerals, organic materials (some naturally occurring, others man-made) plus any materials used to transport or store water. ⁽²⁾

For more than 30 years there has been remarkable growth in the need for quality water purification by all categories of users – municipal, industrial, institutional, medical, commercial and residential. The increasingly broad range of requirements for water quality has motivated the water treatment industry to refine existing techniques, combine methods and explore new water purification technologies. ⁽²⁾

Water has always been seen as the symbol of power and clarity of movement and regeneration for life itself. That’s why people have been seeking methods to improve the taste and odor of drinking water since as

early as 4000 B.C. Ancient Sanskrit and Greek writings recommended water treatment methods such as filtering through charcoal, exposing to sunlight, boiling and straining. ⁽³⁾

Early water treatments were driven by the desire to reduce the visible cloudiness of water, as well as its objectionable taste and appearance. As early as 1500 B.C., the Egyptians reportedly clarified water using the coagulant alum, a chemical that causes suspended particles to settle out of water. During the 1700s, filtration was established as an effective means of removing particles from water, although they had no way of really testing how much. By the early 1800s, slow sand filtration was beginning to be used regularly in Europe, mainly to improve water's taste and odor. By the late 1800s and early 1900s, slow sand filtration was used by water systems in some U.S. cities such as Philadelphia. ⁽³⁾

1.2 Treatment Methods

There are three main classes of treatment processes (summarized below). Typical operation ranges of which are shown in Fig. 1-1.

- (1) Physical processes, which essentially depend on physical properties of the impurity material, e.g. particle size, specific gravity, viscosity etc. Typical examples of this type of process are screening, sedimentation, filtration, and gas transfer.
- (2) Chemical processes, which depend on the chemical properties of an impurity or utilize the chemical properties of added reagents. Examples of chemical processes are coagulation, precipitation, ion exchange and adsorption.
- (3) Biological processes, which utilize biochemical reactions to remove soluble or colloidal impurities, are usually organics. Aerobic biological processes include biological filtration and

activated sludge. Anaerobic oxidation processes are used for the stabilization of organic sludges and high strength organic wastes. ⁽⁴⁾

In many cases all the three processes may be required, in others one or two methods may be combined into a practical and economical plant to achieve the desired result. ⁽⁵⁾

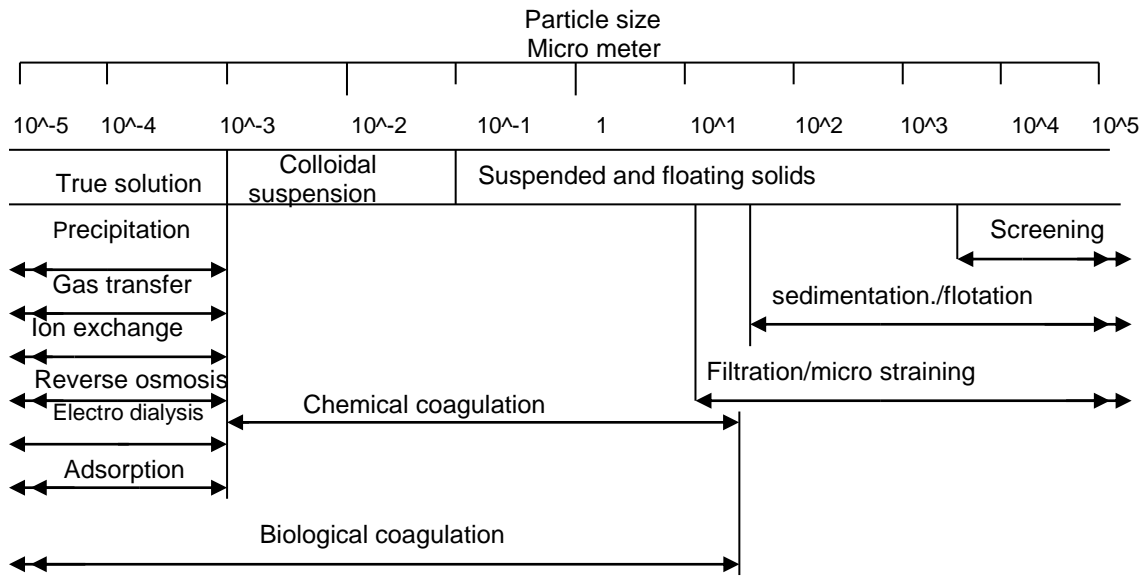


Fig. 1-1: Application of the Main Treatment Processes. ⁽⁴⁾

1-3 Water Pollution

Water pollution is any human-caused contamination of water that reduces its usefulness to humans and other organisms in nature. Pollutants such as herbicides, pesticides, fertilizers, and hazardous chemicals can make their way into our water supply. When our water supply is contaminated, it is a threat to human, animal, and plant health unless it goes through a costly purification procedure. ⁽⁶⁾

Examples of pollution and its effects on water bodies are listed below:

1- Pollutants come from a specific source such as a pipe that discharges used water or other material from a factory into a water body. Such

discharges can harm the aquatic ecosystem. Number of chemical pollutants like chromium oxide and mercury oxide has been found in Iraqi rivers.

2- Pollutants can also come from large areas such as agricultural fields that have been covered with fertilizer or pesticides. Fertilizer and pesticide residues can run off or wash into streams and rivers or seep into soil, contaminating underlying groundwater.

3- Pollutants can also come from parking lots, gardens, driveways, sidewalks, lawns, and roads. Rain water or melted snow can transfer materials such as oil, litter, fertilizers, and salt down storm sewer inlets found on the streets. In some areas, the storm sewer transports this polluted water to a water treatment facility. In other areas, the storm sewer transports this polluted water to a nearby river, lake, stream, or wetland.

4- Pollutants can contaminate our drinking water sources, reduce oxygen levels which can kill fish and other wildlife, accumulate in the tissue of fish we catch and eat from the lakes, and reduce the beauty of the water.⁽⁶⁾

Adsorption techniques for wastewater treatment have become more popular in recent years owing to their efficiency in the removal of pollutants. Adsorption can produce high quality water while also being a process that is economically feasible.⁽⁷⁾

Decolorization is a result of two mechanisms-adsorption and ion exchange, and is influenced by many factors including dye/sorbent interaction, sorbent surface area, particle size, temperature, pH and contact time.⁽⁸⁾

The main adsorbent used in industry is activated carbon. Different physical forms of activated carbon are produced depending on their application.⁽⁸⁾

1-4 Literature Review

Walker and Weatherley (1982) measured the kinetics of acid dye adsorption onto a granular activated carbon. They reported that a single value of a solid diffusivity could be used to describe the adsorption decay curves for constant mass contacting system. However a decrease in solid diffusivity was required for systems with different initial dye concentration. Chemisorption was suggested as the likely mechanism for the acid dye removal from water. The adsorption of dye into carbon can depend upon surface charge on the carbon in the presence of water. Hence, activated carbon will demonstrate a high capacity for both acid and basic dyes⁽⁹⁾

Netzer and Hughes (1984) studied the adsorption of copper, lead and cobalt by carbon. They used an initial concentration of 10 mg/L for each metal to evaluate and optimize process variables like pH, equilibrium, time, and carbon type and carbon dosage. They found that when two or three metals were present in solution there seemed to be a competition for adsorption sites. The required time to complete adsorption was affected by the ratio of metal species to adsorption sites. Lead adsorption was hindered by the presence of other metals with copper having a greater hindrance effect on lead than cobalt. Also approximately twice as much lead as copper was removed and 10 times more lead was removed than cobalt. An optimum pH of 4 was obtained for the two metals.⁽¹⁰⁾

Tan and Teo (1985) studied the combined effect of carbon dosage and initial concentration on the adsorption isotherm of lead and chromium onto activated carbon. They found that the Freundlich and Langmuir equations were unable to adequately describe the observed isotherm under combined influence of carbon dosage and initial adsorption concentration. This had to be modified. The activated carbons used were pre-conditioned by soaking in NHCl solution for twenty four

hours, washed and then soaked in deionized water for twenty four hours before it was oven dried at 103°C. The contact times for the adsorption studies were 3 days. They concluded from their studies that adsorption of lead and chromium by activated carbon showed significant dependency on pH, carbon dosage and initial adsorbate concentration.⁽¹¹⁾

Allen et al. (1989) considered the competitive adsorption of three basic dyes. The results showed that the adsorption capacity of an individual dye decreased in the presence of a second or a third dye. The reduced capacities can be attributed to a number of factors including: interaction between the dyes in solution; change of the adsorbent surface charge due to adsorption; competitive adsorption between the dyes for active sites on the carbon surface where displacement effects replace the other dyes from the adsorption sites. At the beginning of the adsorption process, ramazol yellow is the first to react with the surface of the activated carbon. This in turn may change the surface charge and surface chemistry of the carbon which will decrease the surface sites available to the remaining two dyes in the mixture.⁽¹²⁾

Wilczak and Keinath (1993) studied the kinetics of sorption and desorption of copper (II) and lead (II) on activated carbon. It was found that the sorption of lead (II) and copper (II) on activated carbon consisted of a rapid initial uptake followed by a slow approach to equilibrium which spanned over a period of several weeks. They found that sorption of copper and lead ions on activated carbon were fully reversible.⁽¹³⁾

Choy et al. (1999) considered the adsorption of acid dyes onto granular activated carbon and presented an analysis of the equilibrium adsorption in single component and multicomponent solutions. The data were analyzed for three dyes using the Langmuir, Freundlich, Redlich-Peterson, Tempkin and Dubinin-Radushkevich isotherms. In their work, the Redlich-Peterson isotherm was demonstrated to provide the

correlation for the sorption process. For the multicomponent isotherm models, an extended Langmuir model proved to be successful in predicting the binary equilibrium data.⁽¹⁴⁾

Al Dega et al. (2000) investigated the effect of carbon surface chemistry on the adsorption of three reactive dyes in aqueous solution. In this work, the adsorption capacities for anionic reactive dyes, namely Ramazol Golden Yellow RNL (Reactive Orange107), Ramazol Red RB (Reactive Red 198) and Ramazol Black B (Reactive Black 5) were determined using Filtrasorb 400 activated carbon. Under the same condition, the adsorption capacities decrease as in the following manner: Ramazol Yellow > Ramazol black > Ramazol Red. The adsorption capacity for reactive dyes increased with decrease in the carbon particle diameter. Also he indicated that the adsorption capacity for Ramazol dyes on granular activated carbon f-400 was reduced upon mixing in multi-component solutions. The results also show that carbon affinity was still higher for ramazol yellow followed by ramazol black and is lowest for ramazol red.⁽¹⁵⁾

Meshko et al. (2001) examined the adsorption of two basic dyes, Maxilon Schwarz FBL-01 (MS-300) and Maxilon Goldgelb GLEC (MG-400), from aqueous solution by granular activated carbon and natural zeolite in batch processes and demonstrated that the activated carbon has a much stronger affinity for the dyes than the zeolite by a factor of two.⁽¹⁶⁾

Al-Mas'udi (2005) determined and compared the removal efficiency of color (caused by Al-Hilla Textile Company wastewaters), by using two kinds of adsorbents, namely activated carbon and activated bauxite and six different dyes (reactive; red, blue, yellow and disperse; red, blue, yellow) which are mainly used in the company mentioned above. These dyes are tested in their aqueous solutions to study the different variables

affecting the adsorption behavior on the two kinds of adsorbents. He found that the adsorption rate and, therefore the adsorption capacity of the activated carbon are greater than that for the activated bauxite (255×10^{-5} kg color/kg carbon) and (133×10^{-5} kg color/kg bauxite) respectively. That is due to the fact that for the same bed height column the weight of the carbon is less than the bauxite because of its smaller bulk density; (bulk densities for the adsorbents used are 350 and 665 kg/m³ for activated carbon and activated bauxite respectively). The adsorption process using activated carbon has a good degree of color reduction, reach 99.7%, and for activated bauxite reach 95%.⁽¹⁷⁾

Hassan (2005) studied the blue dye removal from textile company wastewater by the adsorption process using continuous system (fixed-activated carbon bed), and proved that the equilibrium isotherm for the system of adsorbed blue dye on activated carbon was of favorable type. It was well represented by Langmuir and Freundlich equations. The adsorption process using activated carbon insured an excellent degree of reduction for color with removal efficiency 90 %.⁽¹⁸⁾

Zhimang gu et al. (2005) developed Granular activated carbon-based, iron-containing adsorbents (As-GAC) for effective removal of arsenic from drinking water. Granular activated carbon (GAC) was used primarily as a supporting medium for ferric iron that was impregnated by ferrous chloride (FeCl₂) treatment, followed by chemical oxidation. Sodium hypochlorite (NaClO) was the most effective oxidant. Carbons produced from steam activation of lignite were most suitable for iron impregnation and arsenic removal. They suggested that the impregnated iron was predominantly in amorphous form. As-GAC could remove arsenic most efficiently when the iron content was approximately 6%. Further increase of iron decreased arsenic adsorption. The removal of arsenate occurred in a wide range of pH as examined from 4.4 to 11. The

efficiency was decreased when pH was higher than 9.0. The presence of phosphate and silicate could significantly decrease arsenate removal at $\text{pH} > 8.5$, while the effects of sulfate, chloride, and fluoride were minimal. Column studies showed that both As(V) and As(III) could be removed to below $10 \mu\text{g/L}$ within 6000 empty bed volume when the groundwater containing approximately $50 \mu\text{g/L}$ of arsenic was treated.⁽¹⁹⁾

1-5 Aim of study

The objectives of this study are:

- 1- Manufacture solid block activated carbon filters from activated carbon (AC) and local raw material represented by high density polyethylene (HDPE).
- 2- Using this filters for treating water from heavy metals such as Chromium and from colored materials such as tea dye and estimate the efficiency of this filter in removing these contaminants.

Chapter Two

THEORETICAL ASPECT

2-1 Raw Materials

2-1-1 The Element Carbon

The word carbon is derived from the Latin “carbo”, which is according to the Romans meant charcoal (or ember). In the modern world, carbon is, of course, much more than charcoal. From carbon come the highest strength fibers, one of the best lubricants (graphite), the strongest crystal and hardest material (diamond), an essentially non-crystalline product (amorphous carbon), one of the best gas adsorbers (activated charcoal), and one of the best helium gas barriers (amorphous carbon). A great deal is yet to be learned and new forms of carbon are still being discovered such as the fullerene molecules and the hexagonal polytypes of diamond. These very diverse materials, with such large differences in properties, all have the same building block—the element carbon—which is the thread that ties the various constituents and gives it unity.⁽²⁰⁾

2-1-2 Carbon polymorphs (or allotropes)

The extraordinary ability of the chemical element carbon to combine with itself and other chemical elements in different ways is the basis of organic chemistry and of life. This chemical versatility also gives rise to a rich diversity of structural forms of solid carbon. The very wide range of carbon materials is now available with emphasis on chemical bonding and microstructure. The materials reviewed include:

- (i) Crystalline forms of carbon: diamond, graphite, Fullerenes and carbynes.
- (ii) Amorphous carbon films and diamond films.
- (iii) Carbon nanoparticles, including carbon nanotubes.

(iv) Engineering carbons with mirror-less disordered microstructures based on that of graphite. ⁽²¹⁾

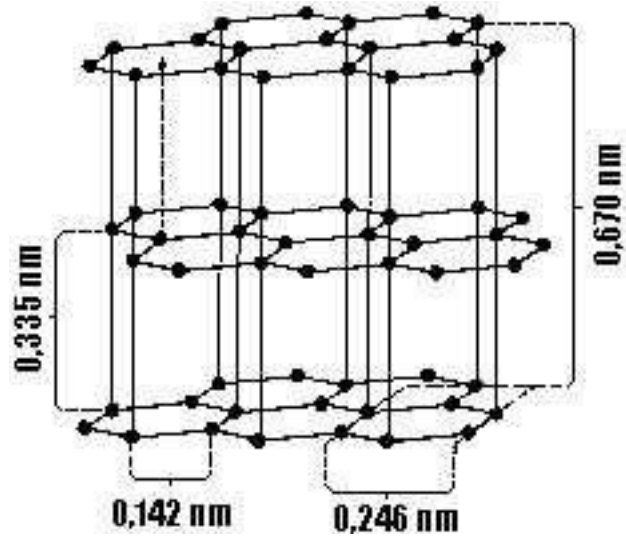
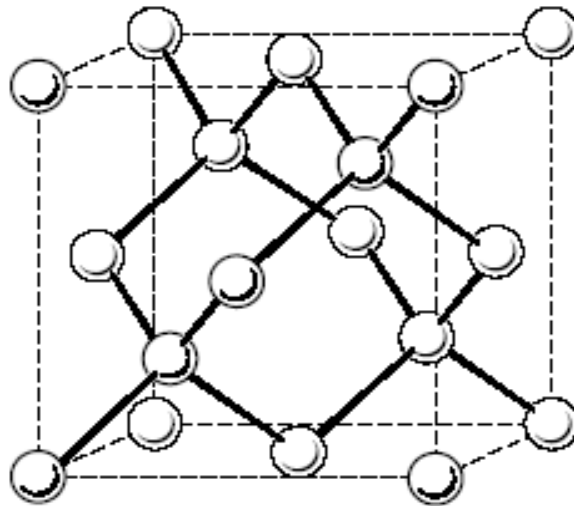
(i) Crystalline Forms of Carbon

The commonest crystalline forms of carbon, F.C.C diamond and hexagonal graphite, are classical examples of allotropy that are found in every chemistry textbook. Both diamond and graphite also exist in two minor crystallographic forms: hexagonal diamond and rhombohedral graphite. To these must be added carbynes and Fullerenes, both of which are crystalline carbon forms. Fullerenes are sometimes referred to as the third allotrope of carbon. The structures of these crystalline forms of carbon are shown in figure 2-1(a & b). ⁽²²⁾

(ii) Carbon Films

The transformations between one form of carbon and another can be classified as follows:

- a) solid-solid transformations, as between diamond and graphite at points 'B' and 'C' in Fig. 2-2.
- b) solid-solute-solid transformations as in the catalyzed transformation of graphite to diamond at 'A' in Fig.2-2.
- c) solid-liquid-solid transformations; these occur when solid carbon phases are flash heated to temperatures above the melting line for the solid phase.
- d) solid-gas-solid transformations in which the product form of carbon is produced by condensation of gaseous carbon species produced by evaporation from the reactant form.

(A) The crystal structures of: hexagonal graphite**(B) The crystal structures of: cubic diamond****Fig. 2-1: structures of carbon.**⁽²²⁾

The last process is important in the production of carbon films. Gaseous carbon species can be produced from solid carbon phases by electric arc-induced evaporation, by other forms of plasma-assisted and laser-induced evaporation, and also by electron and ion beam sputtering. Metastable carbon films can be produced from the gaseous phase, particularly under conditions of rapid cooling or quenching.⁽²¹⁾

(iii) Carbon Nanoparticles

In addition to diamond and amorphous films, nanostructural forms of carbon may also be formed from the vapor phase. Here, stabilization is achieved by the formation of closed shell structures that obviate the need for surface heteroatoms to stabilize dangling bonds, as it is the case for bulk crystals of diamond and graphite. The new-classical example of closed-shell stabilization of carbon nanostructures is the formation of C_{60} molecules and other fullerenes by electric arc evaporation of graphite as in figure 2-3.

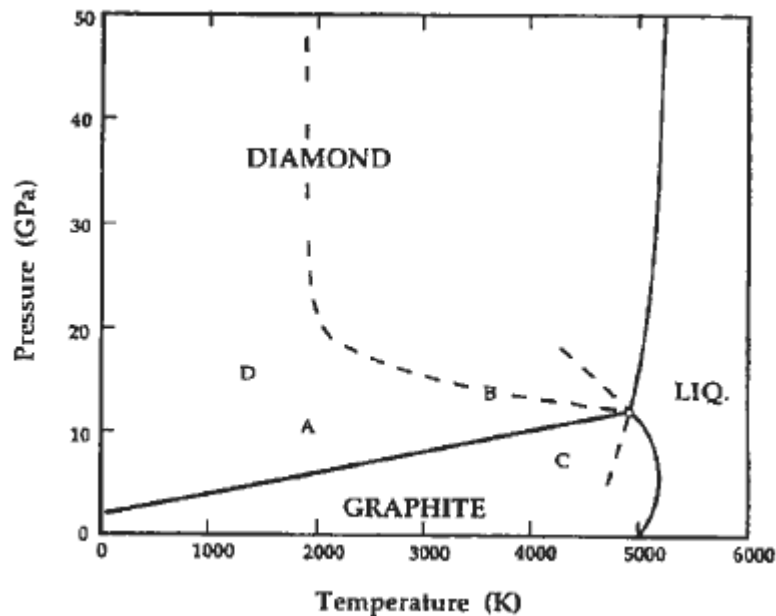


Fig. 2-2: The phases and transition diagram for carbon.⁽²¹⁾

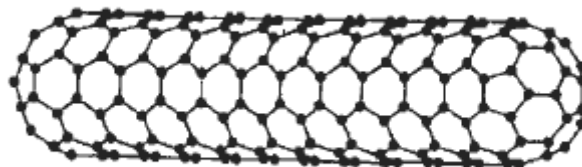


Fig. 2-3: Structure of a single wall nanotube.⁽²¹⁾

(iv) Engineering Carbons

The great majority of engineering carbons have graphitic microstructures or disordered graphitic microstructures. Also, most engineering carbon materials are derived from organic precursors by heat treatment in inert atmospheres (carbonization). A selection of technically important carbons obtained from solid, liquid and gaseous organic precursors is presented in Table 2-1.

Table 2-1: Precursors for engineering carbons.⁽²¹⁾

<i>Primary precursor</i>	<i>Secondary precursor</i>	<i>Example products</i>
Hydrocarbon		Pyrocarbon, carbon blacks, vapour grown carbon
gases		fibers, matrix carbon
Petroleum		Delayed coke, calcined cock
	Petroleum Pitch	Needle coke, carbon fibers, binder and matrix carbon
	Mesophase pitch	Mesocarbon microbeads, carbon fibers
Coals		semi-cock, calcined coke
	Coal chars	Activated carbons
	Coal tar pitch	Premium cokes, carbon fibers, binder and matrix carbons
	Mesophase pitch	Mesocarbon microbeads, carbon fibers
Polymers	polyacrylonitrile	PAN-based carbon fibers
	Phenolic and furan resins	Glassy carbons, binder and matrix carbons
	polyimides	Graphit films and monoliths
Biomass		Activated carbons

During carbonization the organic precursor is thermally degraded by heat treatment at temperatures in the range (450-1000) °C to form

products that undergo condensation or volatilization reactions, the competition between these processes determining the carbon yield. ⁽²¹⁾

The many forms of so called amorphous carbon such as charcoal, soot and lampblack are all actually microcrystalline forms of graphite. In some soot the microcrystals are so small that they contain only few unit cells of the graphite structure. The physical properties of such materials are mainly depended upon the nature and the magnitude of their surface areas. The finally divided forms which present relatively vast surface area with only partially saturated attractive forces readily adsorb large amounts of gases and solutes from solution. . ⁽²²⁾

In this research we focus on the engineering carbon “Activated Carbon” which is used as matrix material for filters used in this study.

2-1-3 Activated carbon

Activated carbon is a black solid substance resembling granular or powdered charcoal. It is extremely porous with a large surface area, and typically produced from organic precursors such as bamboo, coconut shells, palm-kernel shells, wood chips, sawdust, corncob and seeds. ⁽²³⁾

Activated carbon surface properties are both hydrophobic and oleophilic; that is, they “hate” water but “love” oil. When flow conditions are suitable, dissolved chemicals in water flowing over the carbon surface “stick” to the carbon in a thin film while the water passes on. This process is called adsorption. ⁽²⁴⁾

Pure carbon occurs as crystals of diamond or graphite or as fullerene spheres. The atoms in diamond are arranged as tetrahedral, and the atoms in graphite are arranged as sheets of hexagons. Activated carbon grains consist of random arrays of microcrystalline graphite. Such grains can be made from many different organic substances, but various

grades of coal are most commonly used, because the product is hard, dense, and easy to handle. ⁽²⁵⁾

Activated carbon adsorption is an effective means for reducing organic chemicals, chlorine, lead, and unpleasant tastes and odors in effluent or colored substances from gas or liquid streams. Typically for activated materials, surface areas range from 500-1400 m²/gm. Adsorption can be classically defined as absorption on the surface of the material due to capillary condensation inside the multitude of pores/active sites available. Because organic chemicals are often responsible for taste, odor, and color problems, activated carbon filtration can generally be used to remove such impurities. ⁽²³⁾

Activated carbon has a high adsorption capacity or reactivity which arises from the complexity of the chemical surface groups compared to other surfaces. The surface of carbon materials can contain not one but, at least, five markedly different types of surface groups such as carboxylic, lactonic, phenolic, carbonyl and etheric types. This diversity of surface groups makes the surface chemistry (acid-base character) much more versatile than that of other adsorbents. ⁽²⁶⁾

2-1-4 Activated Carbon Types

It is found in two general forms; granular activated carbon (GAC) and powdered activated carbon (PAC). ⁽²⁴⁾

(A) Granular Activated Carbon (GAC)

Granular activated carbon (GAC) by definition is composed of particles with sizes greater than 0.8 mm, about the size of coarse sand. GAC is generally said to have a larger internal surface area and smaller internal pores, GAC is used in continuous processes in areas like water

treatment (drinking & effluent water), solvent recovery, gold recovery, removal of odor and gases. GAC has some very interesting properties such as an adsorbent it traps various large molecules in its porous inside. ⁽²⁴⁾

(B) Powdered Activated Carbon (PAC)

Powdered activated carbon (PAC), of course is composed of particles smaller than 0.8 mm, PAC is thought to have larger pores and a smaller internal surface area, PAC is used in liquid phase applications primarily for removal of undesired compounds impurities in many types of industries including basic drugs, fine chemicals, glucose, and sugar. Powdered carbon is washed with acid and then, repeatedly with water & dried to minimize impurities. It is used in batch processes. Powdered activated carbon (PAC) cannot be used in a fixed-bed arrangement because of the tiny size of its particles and subsequent high head loss that would result from passing water through it, powdered activated carbon is much more difficult to regenerate than granular in wastewater treatment. ⁽²⁷⁾

2-1-5 Polyethylene

Polyethylene, often called polythene, is probably the plastic most well known to the consumer and is used in greater volume worldwide than any other plastic. ⁽²⁸⁾

Polyethylene is considered one of the most important synthesis polymers and it is produced in many petrochemical compounds in mass scale. ⁽²⁹⁾

Polyethylene, a thermoplastic resin, is the principal member of the polyolefin class of polymers. It was discovered by accident in 1933 when scientists working in the (ICI) laboratories in the UK found that when ethylene was subjected to high pressure a wax-like polymeric substance was formed. The original polyethylene called low density polyethylene

(LDPE) has been supplemented with two other widely-used types –high-density polyethylene (HDPE) and linear low-density polyethylene (LLDPE). Other classifications are sometimes used for particular specialized polyethylene polymers. They include very-low-density polyethylene (VLDPE), medium-density polyethylene (MDPE) – which is often considered to be at the high-density end of (LDPE) polymers – ultra-high-molecular-weight polyethylene (UHMWPE), and cross-linked polyethylene.

As the names of the polyethylene types suggest, the principal physical property marking the differences between them is the density. The differences in density are basically due to differences in the degree of crystallinity, which also influences the plastics' melting point ranges. ⁽²⁸⁾

Specific values of different types of polyethylene are listed in Table 2-2.

(30)

Table 2-2: Variation of polyethylene density. ⁽³⁰⁾

<i>No.</i>	<i>Density (g/cm³)</i>	<i>Common name</i>	<i>Symbol</i>
1	0.885-0.914	Very Low Density PE	VLDPE
2	0.915-0.925	Low Density PE	LDPE
3	0.926-0.940	Medium Density PE	MDPE
4	0.941-0.959	High Density PE	HDPE
5	0.960 and higher	Very high Density PE	VHDPE

HDPE has little branching, giving it stronger intermolecular forces and tensile strength than lower density polyethylene. It is also harder and more opaque and can withstand somewhat higher temperatures (120 °C for short periods, 110 °C continuously).⁽³¹⁾

The repeating mer for polyethylene is shown in figure 2-4.

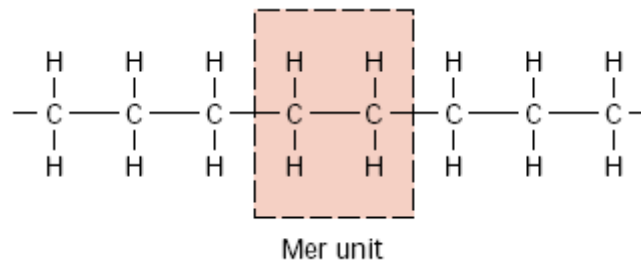


Fig. 2-4: a schematic representation of mer and chain structures for polyethylene.⁽³¹⁾

2-2 Overview of Color Removal Methods

There are many methods for removal of color from the effluent wastewater. These methods can be classified according to the type of the method, as adsorptive, precipitative and reactive. There is a method of classification for the color removal processes as shown in Figure 2-5.⁽³²⁾

The effectiveness of the decolorization techniques can be measured by different methods. These methods can be used to measure the true and apparent colors of the sample. The apparent color of a sample is the perceived color that includes the combined effect of colored material, turbidity and suspended solids. The true color is only due to the dissolved colorant in the sample with the turbidity and suspended matter removed by filtration or centrifuge. Turbidity is seen due to the scattering of light by small particles, suspended matter or air bubbles.⁽³³⁾

Color may occur in drinking water for any one or more of several reasons. It may be due to the presence of colored organic substances originating in the decay or aqueous extraction of natural vegetation, such as in soil runoff; the presence of metals such as iron, manganese, copper

and chromium, which are abundant in nature. They are weathered from rocks or corroded from distribution systems by water, and are naturally colored; or by the presence of highly colored industrial wastes. The most common of which are pulp, paper, leather industry and textile wastes. ⁽³⁴⁾

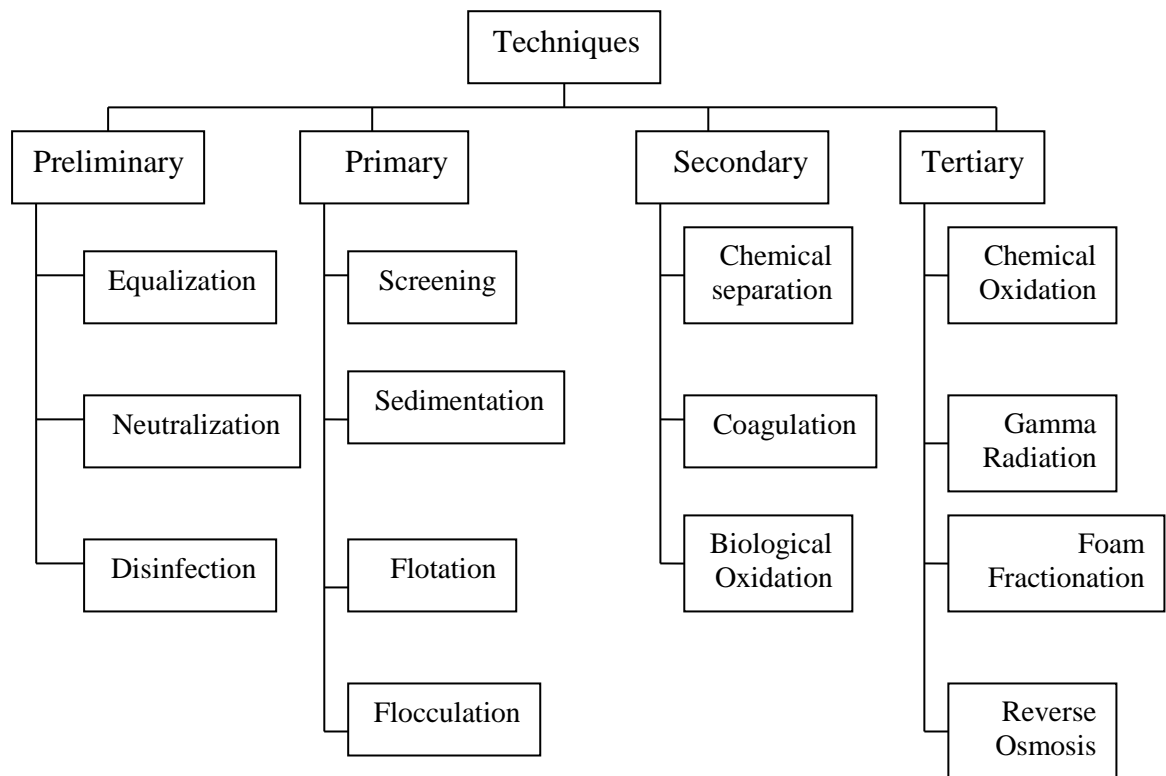


Figure 2-5: Methods of color removal. ⁽³²⁾

2-2-1 Adsorptive Methods

Adsorption is a physiochemical wastewater treatment process that is gaining prominence as a means of producing quality effluents that are low in concentrations of dissolved organics. The adsorptive methods of decolorization use a sorbent medium that physically removes the dye ions and other contaminants from the effluent through physicochemical process of adsorption. In adsorption, the dissolved molecules are attracted to the surface of the adsorbent by physical/chemical forces. However there are many factors that need to be considered while using these

adsorbents. Most important among them are the cost and availability of the sorbents. Disposal of the spent sorbent is also of primary concern.

The most sorbents medium that physically removes the dye are activated carbon which is used in this work, natural wastes, chitosan and synthetic resin.⁽³⁵⁾

2-2-2 Precipitative Methods

The precipitative methods remove color by changing the solubility of the dye such that the dye molecules settle out of the solution or by co-precipitation by sorbing onto an auxiliary precipitative material. Like the adsorptive methods, these methods also involve physical removal of the colored species from water. Most of the precipitative methods include reacting the material with iron hydroxide or using a polymer flocculating material.⁽³⁵⁾

2-2-3 Reactive Decolorization Methods

In the reactive methods of color removal, a part of the chromophore is destroyed that results in a colorless product. Molecules that have conjugated π bonds in their structure can absorb visible light that is re-emitted at the same or different wavelengths. If the reflected or transmitted light is in the visible region, it is detected as color. However when the conjugation is broken into smaller molecules, the dye no longer appears colored. This type of decolorization can involve chemical, biological or radiation methods of color reduction. But most of the new synthetic dyes are produced to withstand such degradation, making this process difficult.⁽³⁵⁾

2-3 Manufacturing of Activated Carbon

Activated carbon has been produced mainly from high carbon content material such as wood, coal, peat, lignin, nutshells, sawdust, bone, and petroleum coke. The production of activated carbon involves two

processes, namely, carbonization, followed by activation. Before carbonization and activation, the starting materials are adjusted to exhibit the desired final physical properties such as granule size, shape, roughness, and hardness. These properties are influenced by production techniques. Blends of the pulverized material and binders (sugar, tar, pitch, and lignin) are often used to obtain desired particle size during extrusion.

Carbonization or pyrolysis consists of slow heating of the material at temperatures usually below 800°C in the absence of air. During this stage of pyrolysis, volatile products are removed from the starting material. Activation consists of treating the pyrolyzed char with activating agents such as steam or carbon dioxide at elevated temperatures that transforms the char into numerous pores which are systematically developed and enlarged, thus enabling the production of a well defined pore system in the activated carbons.

During activation, the surface area or adsorption is determined by (1) the chemical nature and concentration of the activating agent, (2) the temperature of the reaction, (3) the extent to which the activation is conducted, and (4) the amount and kind of mineral ingredients in the char.⁽³⁶⁾

Based upon raw material and intended application, the manufacturing of activated carbon falls into two main categories: thermal activation and chemical activation. In general, thermal activation involves the heating/gasification of carbon at high temperatures, while chemical activation is characterized by the chemical dehydration of the raw material at significantly lower temperatures.⁽³⁷⁾

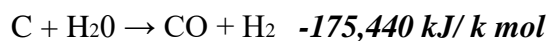
2-3-1 Thermal Activation Processes

Thermal activation is characterized by two processing stages: thermal decomposition or carbonization of the precursor, and gasification or

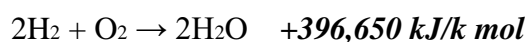
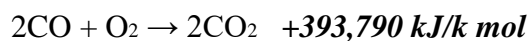
activation of the carbonized char material. In the carbonization step, hydrogen and oxygen are removed from the precursor (raw material) to generate a basic carbon pore structure. During activation, an oxidizing atmosphere such as steam is used to increase the pore volume and particle surface area through elimination of volatile products and carbon burn-off. Thermal activation precursors include coal and coconut shells. Thermal activation is usually carried out in directly fired rotary kilns or multi-hearth furnaces, with temperatures of greater than 1000 °C achieved in process. ⁽³⁸⁾

The following reactions are included in this process:

Initially, gasification of the carbonized material with steam occurs; a reaction known as the Water-Gas reaction:-



This reaction being endothermic, temperature is maintained by partial burning of the CO and H₂ formed:-



Activated carbons produced by steam activation generally exhibit a 'fine' pore structure, ideal for the adsorption of compounds from both the liquid and vapour phases. ⁽³⁹⁾

2-3-2 Chemical Activation Processes

In chemical activation processes, the precursor is first treated with a chemical activation agent, often phosphoric acid, and then heated to a temperature of 450 -700 °C in an activation kiln. The char is then washed with water to remove the acid from the carbon. The filtrate is passed to a

chemical recovery unit for recycling. The carbon is dried, and the product is often screened to obtain a specific particle size range. ⁽³⁸⁾

Activated carbons produced by chemical activation generally exhibit a very 'open' pore structure, ideal for the adsorption of large molecules. ⁽³⁹⁾

2-4 Regeneration of Activated Carbon

Economic studies generally show that it is less costly to regenerate the spent carbon, preferably at the site of the application, than to discard it and use new carbon. Regeneration usually involves removing the adsorbed contaminants from the carbon using temperatures or processes that drive the contaminants from the carbon but that do not destroy the contaminants or the activated carbon. ⁽⁴⁰⁾

As contaminants are adsorbed, the carbon's adsorptive capacity is gradually exhausted. When the carbon's adsorptive capacity is reached, it is considered "spent," and it must be regenerated, reactivated, or disposed of. Although some manufacturers and researchers use the terms "regeneration" and "reactivation" interchangeably, but in fact, "regeneration" means removing the contaminants from the carbon without destroying them and "reactivation," which occurs at very high temperatures, means destroying the contaminants and reactivating the carbon. ⁽⁴¹⁾

2-4-1 Thermal Regeneration Method

Thermal regeneration of granular activated carbon consists of three basic steps; (1) drying, (2) baking of adsorbate and (3) activation by oxidation of the carbon residues from decomposed adsorbates. Drying requires between 100 °C and 700 °C and activation temperature above 750 °C. All of these three steps can be carried out in a direct fired, multiple hearth furnace. This is the best commercial requirement available for regeneration of carbon for use in combination with

wastewater treatment. The capacity of the activated carbon will generally decrease by approximately 10% during the first thermal regeneration and another 10% during the next 5-10 regenerations.⁽⁴²⁾

The manner in which the regeneration is done depends on several factors (1) physical and chemical properties of the pollutant, (2) the media in which the adsorption phase is carried out (gas or liquid), and (3) recovery value of the pollutant.⁽⁴³⁾

2-4-2 Chemical Regeneration

This regeneration method can be carried out with sodium hydroxide provided that high molecular weight colloids are removed before the treatment of the activated carbon. It is possible with this chemical regeneration to recover for example phenols and remove color bodies.

When sodium hydroxide or solvent is used, the adsorbate is passed through the carbon bed in the opposite direction to that of the surface cycle until all is removed. The bed is then drained and the regenerated carbon is ready to go back into the stream.⁽⁴⁴⁵⁾

2-4-3 Biological Regeneration

This method has obvious limitations-like many organic compounds not biodegradable. Further the microorganisms of which the smallest one has a diameter of $10A^{\circ}$ will not be able enter into the internal pore structure of the carbon where most of the organic matter is adsorbed. However, it may have some effects on the adsorption/ desorption equilibria, but the decomposition products will rarely be CO_2 and H_2O , more likely small organic acids and bases that have to be further treated.⁽⁴⁴⁾

2-4-4 Steam Regeneration

If the adsorbate is volatile, recovery may be carried out by passing steam through the carbon bed.

This is usually used in solvent recovery systems in vapor streams. The solvents are typically reused in the process. Usually the steam can achieve greater than 95% recovery /removal efficiency. Very few liquid phase carbon systems are steamed due to the amount of energy and effluent requirements. ⁽⁴⁵⁾

2-4-5 Hot Gas Regeneration

This is used where there are multiple contaminants in a vapor stream and the carbon is used to concentrate them. Once breakthrough happens, a hot gas is used to desorb the contaminants from the carbon. Then they are either condensed out or incinerated. Depending upon the contaminates hot nitrogen, air, CO₂, or combusted products can be used. If chlorinated organics are adsorbed, a scrubber may be necessary. ⁽⁴⁴⁾

2-4-6 Pressure Swing Adsorption

If light organics are adsorbed, a pressure swing adsorption system can be used to adsorb carbons of the contaminates. Condensers are used and the organics are usually recycled back to the system.

There are other regeneration methods but they are for limited application such as, pH swing method which is used to recover the phenol and reuse it, and solvent extraction method. ⁽⁴⁶⁾

2-5 Filtration Mechanism By Activated Carbon

The specific mode of activated carbon action is extremely complex, and has been the subject of much study and debate. Activated carbon has both chemical and physical effects on substances where it is used as a treatment agent. Its activity can be separated into (1) adsorption; (2) mechanical filtration; (3) ion exchange; and (4) surface oxidation.

Adsorption is the most studied of these properties in activated carbon. ⁽⁴⁷⁾

2-5-1 Adsorption Definition

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or, more rarely, a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. The term sorption encompasses both processes, while desorption is the reverse process. ⁽⁴⁸⁾

Adsorption differs from absorption, in that an adsorbed substance remains at the surface while an absorbed substance spreads throughout the absorbing material.

Adsorption is a natural process by which molecules of a dissolved compound collect on and adhere to the surface of an adsorbent solid. It occurs when the attractive forces at the solid surface overcome the attractive forces of the liquid.

It is a term used to describe the existence of a higher concentration of a substance at the interface between a fluid and a solid that is present in the fluid. It can be divided into physisorption and chemisorption. ⁽⁴⁹⁾

A) Physical Adsorption

Physical adsorption is a reversible equilibrium process caused by intermolecular forces (Van der Waal's forces) of attraction between molecules of the solid and the substance to be adsorbed. It is equilibrium coverage similar to surface liquefaction. Coverage proceeds first with adsorption on surface atoms but is quickly followed by generation of additional layers even before complete monolayer forms.

Physical adsorption is usually predominant at low temperature, and is characterized by relatively low heat of adsorption, that is the adsorbate is not held strongly to the adsorbent as for chemical adsorption. The heat generated by physical adsorption is usually less than 42 kJ/mol. ⁽⁵⁰⁾

► Characteristics of physisorption:

- 1- Similar order of magnitude to the latent heats of evaporation.
- 2- Extensive physisorption usually occurs at low temperature.

B) Chemical Adsorption

Chemical adsorption is the result of chemical interaction between the solid and the adsorbed substance. The strength of chemical bond may vary considerably, and identifiable chemical compounds in the usual sense may not actually form, but the adhesive force is much greater than that found in physical adsorption. The process is frequently irreversible, and on adsorption, the original substance will often be found to have undergone a chemical change. The same substance which, under conditions of low temperature, will undergo substantially only physical adsorption upon a solid will sometimes exhibit chemical adsorption at higher temperature, and both phenomena may occur at the same time. The heat liberated during chemical adsorption is usually large (42-210 kJ/mol) it is of the order of the heat of chemical reaction. .⁽⁵⁰⁾

► Characteristics of chemisorption:

- 1- Large enthalpy for adsorption.
- 2- Adlayer is stable to relatively high temperature.

2-5-2 Mechanical Filtration

Mechanical filtration involves the physical separation of suspended solids from a liquid passing through carbon arrayed as a porous media in a column or bed. Any finely divided solid—such as sand or cellulose—can accomplish this. While this accounts for some of the clarification properties of carbon, it is seldom the sole reason for the selection of carbon as a clarification medium. The effectiveness of filtration depends on particle size, bulk density, and hardness. While a smaller particle size results in a clearer liquid, it also slows the speed of processing. Bulk

density determines how much carbon can be contained in a given container. Hardness matters because the articles need to have sufficient strength to block the particulate matter being filtered. ⁽⁵¹⁾

2-5-3 Ion Exchange

Ion exchangers are classified according to (1) their functionality and (2) the physical properties of the support matrix. Cation and anion exchangers are classified in terms of their ability to exchange positively or negatively charged species. Strongly acidic and strongly basic ion exchangers are ionized and thus are effective at nearly all pH values (pH 0–14). Weakly acidic exchangers are typically effective in the range of pH 5–14. Weakly basic resins are effective in the range of pH 0–9. Weakly acidic and weakly basic exchangers are often easier to regenerate, but leakage due to incomplete exchange may occur.

Ion exchange is defined as a ‘reversible chemical reaction between a solid and an aqueous solution that allows the interchange of ions. Coal is a natural ion exchanger. Ion exchange can be enhanced by chemical activation. Carbon surfaces have both negative (anionic) or positive (cationic) charges to attract free ions in solution or suspension, depending on how they are treated. Treatment of carbon with a base increases the capacity of carbon to exchange anions; acidulation of the surface makes carbon a powerful cation exchanger. ⁽⁵²⁾

2-5-4 Surface Oxidation

Surface oxidation involves the chemisorption (=chemical adsorption) of atmospheric oxygen to the carbon and the further reaction of the surface oxides that chemically react with other substances that are oxidized. The surface of activated carbon has an electrical double layer. ⁽⁴⁷⁾

2-6 Pore Structure of Activated Carbon

Three groups of pores can be distinguished in an activated carbon.⁽⁵³⁾

1. Micropores (8-20 Angstrom*)
2. Transitional or Meso-pores (20-500 Angstrom*)
3. Macropores (> 500 Angstrom*)

*One angstrom = 0.0000001 mm.

Figure 2-6 shows a schematic of an activated carbon particle.⁽⁵⁴⁾

The major portion of the surface area is derived from the small diameter micropore and the medium diameter transitional pore regions. Micropores have been found to be the most effective in trapping small molecules in gas and liquid phase applications. The transitional pore region is most suitable for adsorbing large molecular species such as color molecules.⁽⁵³⁾

The raw material for an activated carbon plays a major part in determining the ability of the final product to adsorb certain molecular species. Activated carbons produced from coconut shells exhibit a predominance of micropores; while coal based carbons have a wider range of transitional pores. The development of an extensive macropore structure is found when either peat or wood is used as the raw material.⁽⁵⁵⁾

For carbons with a predominance of micropores, the internal surface area is incredibly large. Many activated carbons have internal areas in the region of 500 to 1,500 square meters per gram, and it is this enormous area which makes them effective adsorbents. Viewed another way, just one pound of activated carbon at 950 m²/g has the equivalent surface area of 100 football fields. All organic compounds will be selectively adsorbed in the activated carbon pores dependent on their size.⁽⁵³⁾

The surface and pores structure of activated carbon shown in Figure 2-7 below.⁽⁵⁶⁾

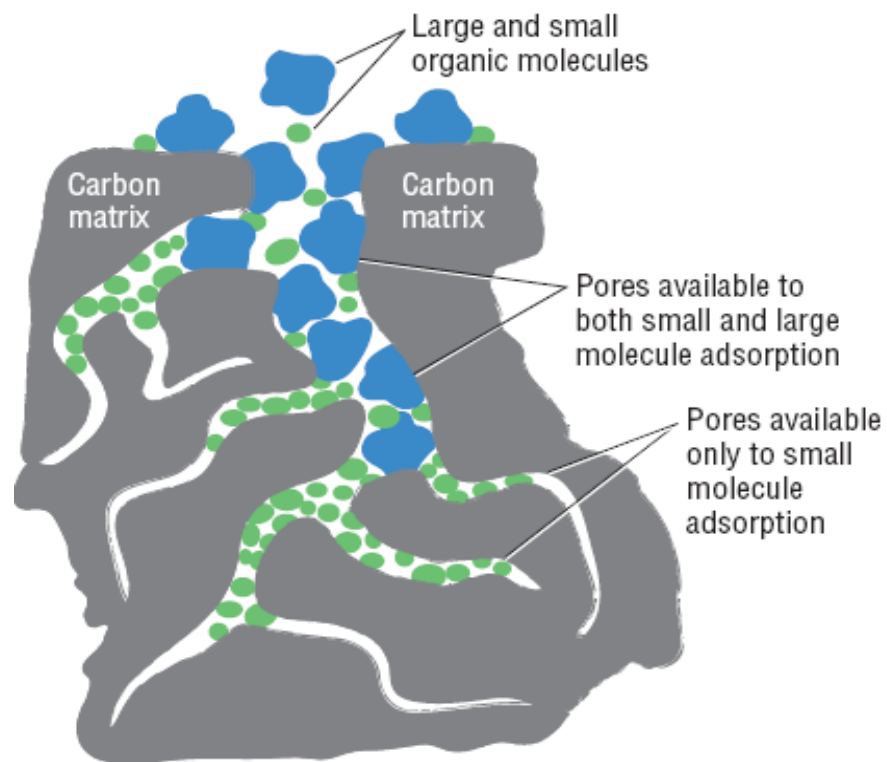


Figure 2-6: A schematic of an activated carbon particle.⁽⁵⁴⁾

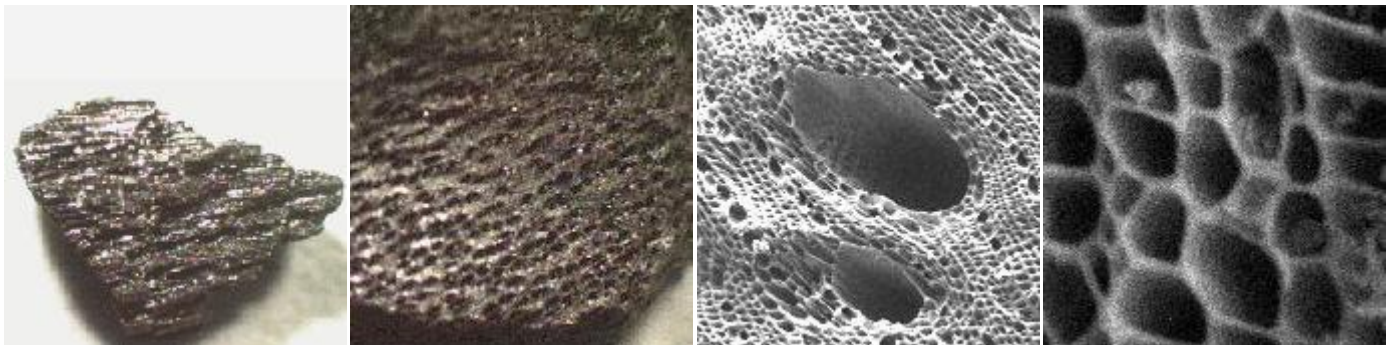


Fig. 2-7: Close-up of Carbon Surface and Pores magnification increases left to right.⁽⁵⁶⁾

The reason that activated carbon is such an effective adsorbent material is due to its large number of cavernous pores. These provide a large surface area relative to the size of the actual carbon particle and its visible exterior surface. An approximate ratio is 1 gram = 100 m² of surface area

(56). These pores of activated carbon are clearly shown by scanning electron microscopy in Figure 2-8.

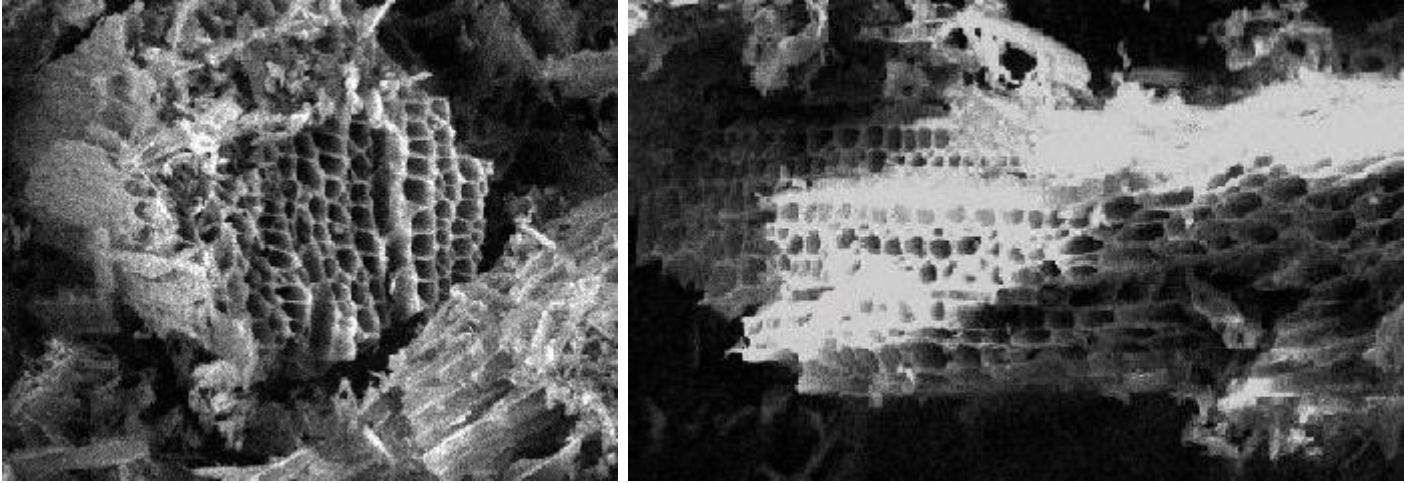


Figure 2-8: Scanning Electron Microscope Images of activated carbon Pores.⁽⁵⁶⁾

2-7 : Efficiency of Activated Carbon Filters to Remove contaminants

Activated carbon (AC) filter is most effective in removing organic contaminants from water. Organic substances are composed of two basic elements, carbon and hydrogen. Because organic chemicals are often responsible for taste, odor, and color problems, AC filter can generally be used to improve aesthetically objectional water. Activated carbon filter will also remove chlorine and traces of heavy metals like mercury and chromium groups. AC filter is recognized by the water quality association as an acceptable method to maintain certain drinking water contaminants within the limits of the National Drinking Water Standards, United States Environmental Protection Agency (EPA) Table 2-3.⁽⁵⁶⁾

Table 2-3. Water contaminants that can be reduced to acceptable standards by activated carbon filter. (Water Quality Association (WQA), 1989)⁽⁵⁶⁾

Primary Drinking Water Standards	
Contaminant	ppm (*MCL)
Inorganic Contaminants	
Organic Arsenic Complexes	0.05
Organic Chromium Complexes	0.05
Mercury (Hg+2) Inorganic	0.05
Organic Mercury Complexes	0.002
Organic Contaminants	
Benzene	0.005
Endrin	0.0002
Lindane	0.004
Methoxychlor	0.1
1,2-dichloroethane	0.005
1,1-dichloroethylene	0.007
1,1,1-trichloroethane	0.200
Total Trihalomethanes (TTHMs)	0.10
Toxaphene	0.005
Trichloroethylene	0.005
2,4-D	0.1
2,4,5-TP (Silvex)	0.01
Para-dichlorobenzene	0.075
Secondary Drinking Water Standards	
Contaminant	(**SMCL)
Color	15 color units
Foaming Agents (MBAS)	0.5 mg/L
Odor	3 threshold Odor number

***Maximum Contaminant Level**

****Secondary Maximum Contaminant Level**

Activated carbon (AC) filter does remove some organic chemicals that can be harmful if present in quantities above the EPA Health

Advisory Level (HAL), including in this category trihalomethanes (THM), pesticides, industrial solvents (halogenated hydrocarbons), polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs).⁽⁵⁷⁾

2-8 Water Contaminants not Removed by AC Filtration

Similar to other types of water treatment, AC filtration is effective for some contaminants and not effective for others. AC filtration does not remove microbes, sodium, nitrates, fluoride, and hardness. Lead and other heavy metals are removed only by a very specific type of AC filter.⁽⁵⁸⁾

2-9 Types of Activated Carbon

Most popular forms of activated carbon used in the treatment of point of use (POU) drinking water filters are granular activated carbon (GAC), extruded solid carbon block (CB) and powdered activated carbon (PAC).⁽⁵⁹⁾

2-9-1 Granular and Powdered Activated Carbon (GAC):

In these types of filters, water flows through a bed of loose activated carbon granules, which trap some particulate matter and remove some chlorine, organic contaminants, and undesirable tastes and odors. There are three main problems associated with GAC filters: channeling, dumping, and an inherently large pore size. Most of the disadvantages discussed below are not the fault of the activated carbon filtration media, rather, the problem is the design of the filters and the use of loose granules of activated carbon.

The advantages of GAC filters include:

1-Simple GAC filters are initially used for aesthetic water treatment, since they can reduce chlorine and particulate matter as well as improve the taste and odor of the water.

2-Loose granules of carbon do not restrict the water flow to the extent of Solid Block Activated Carbon (SBAC) filters. This enables them to be used in situations, like whole house filters, where maintaining good water flow rate and pressure is important.

3-Simple, economical maintenance. Typically an inexpensive filter cartridge needs to be changed every few months to a year, depending on water use and the manufacturer's recommendation.

4-GAC filters do not require electricity, nor do they waste water.

5-Many dissolved minerals are not removed by activated carbon. In the case of calcium, magnesium, potassium, and other beneficial minerals, the taste of the water can be improved and some (usually small) nutrient value can be gained from the water. ⁽⁶⁰⁾

The disadvantages of GAC filters include:

1-Water flowing through the filter is able to "channel" around the carbon granules and avoid filtration.

2-Pockets of contaminated water can form in a loose bed of carbon granules, with changes in water pressure and flow rates, these pockets can collapse, "dumping" the contaminated water through the filter into the "filtered" flow.

3-Since the carbon granules are fairly large (0.1mm to 1mm in one popular pitcher filter), the effective pore size of the filter is relatively

large (20 - 30 microns or larger). GAC filters, by themselves, can not prevent bacteria.

4-Hot water should NEVER be run through a carbon filter
5-Also, if you think of a bed of charcoal that traps an occasional bacterium, picks up a bit of organic material, and removes the chlorine from the water, you can see how these filters might become breeding grounds for the bacteria they trap. You will see warnings about GAC filters suggesting you run water through them for a few minutes each morning to flush out any bacteria. ⁽⁶⁰⁾

2-9-2 Solid Block Activated Carbon (SBAC)

Carbon blocks (CB's) are made of single or various blends of carbons combined with thermoplastic polymers which are pulverized to a fine dust then shaped in various forms under high pressure. The binder can be composed of nearly any thermoplastic materials including , for example, polyolefin's such polyethylene, polypropylene, polyvinyls such as polyvinyl chloride; polyvinyl esters such as polyvinyl acetate; copolymers and block interpolymers. Unlike the other carbon forms, the CB's are industrial grade filters. They are made in various sizes and micron ratings (nominal), are physically strong therefore they do not "channel" nor "collapse" under pressure change, however they "dump" if their media is exhausted. ⁽⁵⁹⁾

Activated carbon is the primary raw material in solid carbon block filters; but instead of carbon granules comprising the filtration medium, the carbon has been specially treated, compressed, and bonded to form a uniform matrix. SBAC, like all filter cartridges, eventually become plugged or saturated by contaminants and must be changed. ⁽⁶⁰⁾

The advantages of SBAC filters include:

1-Provide a larger surface area for adsorption to take place than granular activated carbon (GAC) filters for better contaminant reduction.

2-Provide a longer contact time with the activated carbon for more complete contaminant reduction.

3-Provide a small pore size to physically trap particulates. If the pore size is small enough, around 0.5 microns or smaller, bacteria that become trapped in the pores do not have enough room to multiply, eliminating a problem common to GAC filters.

4-Completely eliminate the channeling and dumping problems associated with GAC filters.

5-SBAC filters are useful in emergency situations where water pressure and electricity might be lost. They do not require electricity to be completely effective, and water can even be siphoned through them.

6-SBAC filters do not waste water like reverse osmosis {Water pressure is

used to force water molecules through a membrane that has extremely tiny pores, leaving the larger contaminants behind. Purified water is collected from the "clean" side of the membrane, and water containing the concentrated contaminants is flushed down the drain from the "contaminated" side}.

7-Many dissolved minerals are not removed by activated carbon. In the case of calcium, magnesium, potassium, and other beneficial minerals, the taste of the water can be improved and some (usually small) nutrient value can be gained from the water.

8-Simple, economical maintenance. Typically an inexpensive filter cartridge needs to be changed every few months to a year, depending on water use. ⁽⁶⁰⁾

● This combination of features provides the potential for greater adsorption of many different chemicals (pesticides, herbicides, chlorine, chlorine byproducts, etc.) and greater particulate filtration of parasitic cysts, asbestos, etc. than many other purification processes available. By using other specialized materials along with specially prepared activated carbon, customized SBAC filters can be produced for specific applications or to achieve greater capacity ratings for certain contaminants like lead, mercury, arsenic, etc. ^(60, 61)

The disadvantages of SBAC filters include:

1-SBAC filters, like all activated carbon filters, do not naturally reduce the levels of soluble salts (including nitrates), fluoride, and some other potentially harmful minerals like arsenic (unless specially designed) and cadmium. If these contaminants are present in the water, reverse osmosis would usually be the most economical alternative followed by distillation.

2-Hot water should NEVER be run through a carbon filter.

3-As SBAC filters remove contaminants from the water they gradually lose effectiveness until they are no longer able to adsorb the contaminants. ⁽⁶⁰⁾

**2-10 The Features and Benefits of Extruded Carbon Filters/
Axial versus Radial Flow**

Figure 2-9 below shows the axial and radial flow through carbon cartridge. ⁽⁶²⁾

Axial flow forces the water through a pre-filter end cap, into a loose granulated activated carbon bed and exits as treated water. The limitations of this design are profound. GAC filters consist of loose beds of particles packed into a non-rigid plastic tube. This plastic container often expands away from the carbon when under pressure. As a result bypassing and channeling can occur. Another huge limitation is made apparent by its given name granulated activated carbon. This carbon is granulated (think sand texture) its mesh size and adsorptive capacities are severely restricted. In short, the design definitely restricts the performance.

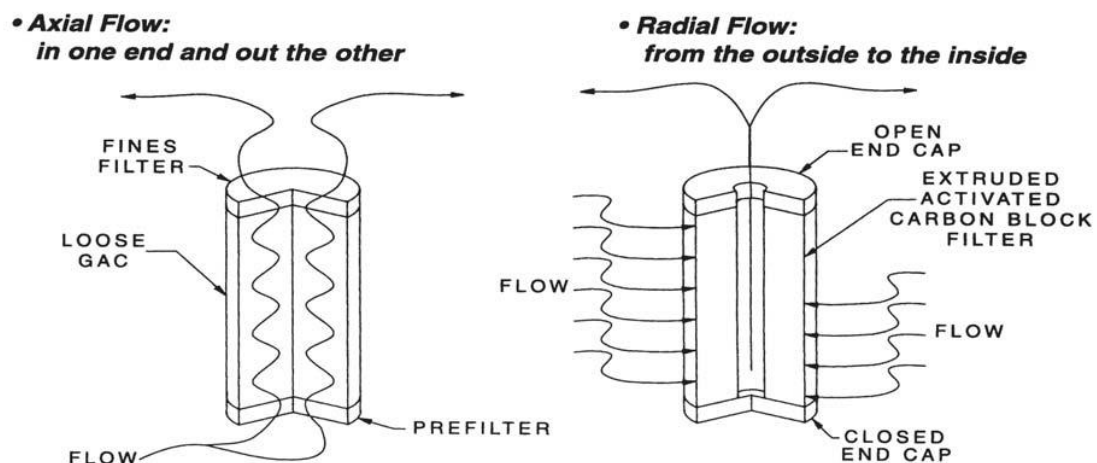


Fig. 2-9: Axial versus Radial Flow.⁽⁶²⁾

Radial flow allows water to flow from the entire outside of the filter through to the core providing 15 times more surface area than axial flow designs. With this increased surface area. The water moves slowly through a consistent solid carbon block membrane but the flow rates are commensurate with GAC filters because of greater exposed surface areas. Thus can creatively design carbon membranes capable of removing or

reducing lead, volatile organic compounds, chlorine, chloramines, asbestos, cysts and turbidity. These membranes are covered with one or more layers or melt-blown and spun-bounded polypropylene filter media. This exterior surface and the adsorptive membrane can provide filtration to a 0.5 micron nominal rating.⁽⁶³⁾

2-11 Factors Influencing Adsorption

There are many factors affecting the adsorption process including the packed bed height, temperature, pH, influent concentration, flow rate, particle size of adsorbent, surface area of adsorbent, adsorbate molecular weight and mixing of more than one adsorbate.⁽⁴⁾

2-11-1 Packed Bed Height

The main effect of the bed height in the adsorption process is on the capacity of adsorbent. Since increasing the bed height will provide extra particles and additional surface area there will be a remarkable increase in the adsorbent capacity. In addition to that increase in the bed height there will be an increase in the contact time between the pollutant and adsorbent particles that will provide enough period of time for pollutants to penetrate the particles of adsorbent.⁽⁶⁴⁾

2-11-2 Temperature

Because adsorption reactions are usually exothermic, high temperatures would seem to inhibit or slow adsorption, but this is not usually found to be a factor in most systems. An explanation for this may be revealed by considering the rate limiting factor for adsorption again. In carbon, adsorption is limited primarily by the diffusion of solute into the carbon particle. Higher temperatures may impede adsorption at the adsorption site, but they significantly speed up the pace of diffusion, offsetting any negative temperature effect.

Normally the adsorption reactions are exothermic, which means that the adsorption will increase with decreasing temperature, although small variations in temperature do not tend to alter the adsorption process to a significant extent.⁽⁶⁵⁾

2-11-3 Surface Area of Adsorbent

The extent of adsorption is proportional to the surface area (which is defined as that portion of the total surface area that is available for adsorption per unit of adsorbent). This means that the adsorption capacity of nonporous adsorbent should vary inversely with the particle diameter while in the case of highly porous adsorbents, the capacity should be almost independent of the particle diameter.⁽¹⁷⁾

2-11-4 pH

pH strongly influences the adsorption as hydrogen and hydroxyl ions are adsorbed and the charge of the other ions are influenced by pH of the solution. For typical organic pollutants from industrial wastewater the adsorption increases with decreasing pH. Adsorption usually increases as pH and temperature decrease. Chemical reactions and forms of chemicals are closely related to pH and temperature. When pH and temperature are lowered many organic chemicals are in a more adsorbable form.⁽⁶⁶⁾

Undoubtedly, the pH value of the dye solution plays an important role in the whole adsorption process and particularly on the adsorption capacity. Any oxide surface creates a charge (positive or negative) on its surface. This charge is proportional to the pH of the solution which surrounds the oxide particles. A convenient index of the propensity of a surface to become either positively or negatively charged as a function of pH is the value of the pH required to give zero net surface charge. This value is called point of zero charge (pH_{ZPC}). pH_{ZPC} is a critical value for

determining quantitatively the net charge (positive or negative) carried on the activated carbon surface during adsorption of reactive dyes.⁽⁶⁷⁾

2-11-5 Adsorbate Concentration

The initial concentration is inversely related to adsorption equilibrium time. This may be explained by the fact that since the rate of diffusion is controlled by the concentration gradient, it takes a longer contact time to reach adsorption equilibrium for the case of low values initial solute concentration.

Because adsorption rate is limited by diffusion, variables that influence diffusion have a significant effect on adsorption rate. For example, a higher concentration gradient across the surface of the adsorbent particle will increase the rate of adsorption.⁽¹⁷⁾

2-11-6 Flow rate

The effect of flow rate on the adsorption process is very important. This effect is based on the assumption that film diffusion is rate limiting in early portion of column. Increasing the flow rate in this region may be expected to make a compression or reduction of the surface film. Therefore, this will decrease the resistance to mass transfer and increase the mass transfer rate. Also, because the reduction in the surface film is due to disturbance created when the flow of the influent increases resulting from the easy passage of pollutants through the particles and entering easily to the pores.⁽⁶⁸⁾

2-11-7 Adsorbate Solubility

The nature of adsorbate also influences the adsorption. Inversely the relation between the extent of adsorption of solute and its solubility in the solvent (water) from which adsorption occurs. It is generally agreed on that a solute's aqueous solubility is inversely related to adsorption onto adsorbent.

The water solubility of organic compounds within a particular chemical class decreases with increasing chain length, because the compound becomes more hydrocarbon-like as the number of carbon atoms becomes greater. Thus, adsorption from aqueous solution increases as homologous series is ascended, largely because the expulsion of increasingly large hydrophobic molecules from water permits an increasing number of water-water bonds to reform.

Molecular size is of significance if the adsorption rate is controlled by antiparticle transport, in which case the reaction generally proceeds more rapidly than the smaller the adsorbate molecule. Large molecules of one chemical class sorb more rapidly than smaller ones of another if higher energies (driving forces) are involved.⁽¹⁷⁾

2-11-8 Size of Adsorbent Particle

In practice, the primary variable influencing adsorption rate is the size of the carbon particle. Smaller adsorbent particles have a greater ratio of surface area to volume, making them more accessible to diffusion from solution. Notably, the size of the particle has little effect on adsorptive capacity as expressed by total surface area. While it is well known that smaller particles of a given mass of carbon would have a greater surface area than larger particles, the huge internal surface area of carbon dominates the calculation so much that the particle size essentially has no effect on capacity.⁽¹⁷⁾

2-12 Properties of Used Contaminants

In this work we have used the following contaminants:-

1- Chromium Hexavalent Oxide

Elemental chromium is a transition-group metal belonging to group 6 of the periodic table and has oxidation states ranging from 2+ to 6+, of which the divalent, trivalent, and hexavalent forms are the most

important. Elemental chromium does not occur naturally in the environment. The divalent (chromous) state is readily oxidized to the more stable trivalent (chromic) state. Although the Hexavalent (including chromates) state is more stable than the divalent state, it is rarely found in nature. Chromium (6^+) compounds are strong oxidizing agents and are highly corrosive. In the environment, they generally are reduced to chromium (3^+) compounds. The chromium (6^+) compounds most commonly encountered in industry are calcium chromate, chromium trioxide, sodium chromate and dichromate, potassium chromate and dichromate, lead chromate, strontium chromate, and zinc chromate. Chromium(6^+) compounds are widely used as corrosion inhibitors, in the manufacture of pigments, in metal finishing and chrome plating, in stainless-steel production, in leather tanning, and in wood preservatives, Chromium(6^+) compounds also are used in textile dyeing processes, printing inks, drilling muds, pyrotechnics, water treatment, and chemical synthesis.⁽⁶⁹⁾

Most compounds of chromium are colored, the color of chromium oxides shows in the Table 2-2 below.⁽⁷⁰⁾

Table (2-4): The color of chromium oxides.⁽⁷⁰⁾

Formula	Colour
CrO ₃	Deep red
Cr ₃ O ₈	-
Cr ₂ O ₅	-
Cr ₅ O ₁₂ etc	-
CrO ₂	Brown-black
Cr ₂ O ₃	Green

2- Tea Dye

As the most potent chemical component in coffee or tea, is caffeine, below some facts about caffeine: ⁽⁷¹⁾

- The full chemical name for caffeine is 1, 3, 7-trimethylxanthine and its chemical formula is $C_8H_{10}N_4O_2$.
- Caffeine was first isolated from coffee in 1820.
- In its pure state, caffeine is a crystalline white powder.
- Caffeine can be found in 60 different plants.
- 10g of caffeine is considered a lethal dose.
- it's the most popular drug in the world. Ninety percent of Americans consume it in some form every day.
- Over 450,000,000 cups of coffee are consumed in the USA every day!
- Darkly roasted coffee has less caffeine than lightly roasted.
- Contrary to popular belief, caffeine (or coffee) won't help someone sober up if they have had too much to drink. Figure 2-10 shows the structure of caffeine. ⁽⁷²⁾

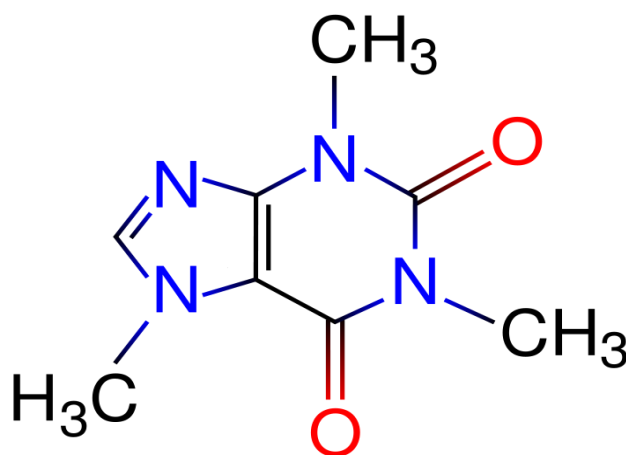


Fig 2-10: structure of caffeine. ⁽⁷²⁾

2-13 Water filter properties

Water filters have many properties. Their importance depends on the purpose that filters used for. For example some types of filters used for mechanical separation so they should have resistance to the friction and deformation and other types used for adsorb some kinds of materials from solution such as heavy metals from waste water.

In this paragraph we explain some physical and mechanical properties in water filters which are assigned this study.

2-13-1 Physical properties:-

1- Apparent Porosity:-

There are two types of porosity, true porosity (which represents the ratio of the volume of opened and closed pores to the total volume of the ceramic body) and apparent porosity (which represents the ratio of the volume of open pores to the total volume of the ceramic body).⁽⁷³⁾

Water absorption is the relation between absorbed water weights in the pores to the weight of solid part of the body.⁽⁷³⁾

The assumption is made that 1 cm³ of water weighs 1 g. This is true for distilled water at room temperature.

The calculation of exterior volume (V) in cubic centimeters is as follows:

$$V = M - S \dots\dots\dots (1-2)$$

The apparent porosity (P) expresses in percentage, the relationship of volume of open pores of the specimen to its exterior volume.

The calculation of apparent porosity is as follows:

$$P = \{(M - D)/V\} * 100\dots\dots\dots (2-2)$$

The water absorption (A) is expresses in percentage, its relationship of the mass of water absorbed to the mass of the dry specimen.

Calculation of water absorption is as follows:

$$A = \{(M - D)/D\} * 100 \dots \dots \dots (3-2)$$

Where:

D: Dry weight (g).

S: Suspended weight in water (g).

M: Saturated weight (g).

V: Exterior volume (cm³).

Many porous media are made of a mixture of discrete large and small grains or particles that are either loose (as dune sand) or held together by compression and/or cementing material (e.g., sandstone). The quantity of finer particles has a marked effect on the porosity. The porosity of consolidated materials depends mainly on the degree of cementation. The porosity of unconsolidated materials depends on the packing of the grains, their shape, arrangement and size distribution.⁽⁷⁴⁾ Porosity and water absorption are two conjugate properties affected by two major factors which are the condition of industrialization process and the pores form inside the ceramic body.

2- Density:-

The density of a material has been defined as its mass (weight) over its volume. For a vitreous objects there are only one weight and one volume involved. For a porous solid, however, there are different ways of expressing its volume, and these must be defined before any precise meaning can be given to its density.⁽⁷³⁾

According to that there are two well known types of density in porous body:

- a) True density: refers to solid part of the body neglecting the pores. It represents the weight of solid material over its real volume.
- b) Apparent density: refers to mass of the body (dry weight) over the total volume (sometimes referred to as the Bulk density).

The bulk density (B), in grams per cubic centimeter, of a specimen is the quotient of its dry mass divided by the exterior volume, including pores. Bulk density will be calculated as follows:

$$B = D/V \dots \dots \dots (4-2)$$

True density will be calculated as follows:

$$T = D / (D - S) \dots \dots \dots (5-2)$$

B: Bulk density (g/ cm³).

T: True density (g/ cm³).

3- Permeability:-

It is one of the physical properties which is an average (or macroscopic) medium property that measure the ability of the porous medium to transmit fluid through it. ⁽⁷⁴⁾

In 1856, Henry Philibert Gaspard Darcy first developed the equation to describe fluid flow through a porous media.

$$Q = - (KA_r/\mu) * (dP_r/dx) \dots \dots \dots (6-2)$$

Q = Volumetric Flow (cm³/s)

K = permeability (Darcy's), (cm² x cP)/(sec.x atm))

A_r = cross-sectional area (cm²)

μ = viscosity (centipoises (cP))

P_r = pressure (atm)

x = length (cm)

$$Q = \frac{cm^3}{s} = \frac{\frac{cm^2 cP}{s \cdot atm} \cdot cm^2 \cdot atm}{cP \cdot cm}$$

There are four conditions that are required for this equation to be valid:

1. Creeping flow – The Reynolds's number based on superficial velocity must be on the order of 1.
2. The porous media is not reactive with the flowing fluid.
3. No accumulation.
4. Single-phase flow

Using this equation, it is possible to determine the permeability (K) of the porous media. ⁽⁷⁵⁾

Filter permeability should be measured under condition close to the practice condition.

4- Maximum pore diameter:-

Max. pore diameter_ the diameter in micrometers of a capillary of circular cross section that is equivalent (with respect to characteristics related to surface tension effects) to the largest pore in the filter under consideration.

Maximum Pore Diameter is determined by immersing the filter in a suitable test liquid and applying air pressure until the first bubble of air passes through the filter. The maximum pore diameter is calculated from the surface tension of the test liquid and the applied pressure. ⁽⁷⁶⁾

Procedure for measuring maximum pore diameter can be done according to ASTM standard (E-128-99) as the following: ⁽⁷⁶⁾

Thoroughly wet the clean filter to be tested by soaking it in the prescribed test liquid. Connect the filter to a controllable source of clean, dry compressed air, and a manometer. Immerse the filter just below the

surface of the test liquid and gradually increase the air pressure at a rate of about (5 mmHg/min) in the area of the test until the first dynamic bubble passes through the filter and rises through the liquid. The appearance of the first true dynamic bubble is readily recognized since it is followed by a succession of additional bubbles. Read the pressure from the manometer. If the test is to be repeated, thoroughly re-wet the filter by soaking it in the test liquid, before proceeding with the retest.

Calculate the maximum pore diameter from the following equation: ⁽⁷⁶⁾

$$D_{\max.} = 30\gamma/P_r \dots\dots\dots (7-2)$$

Where:

$D_{\max.}$ = maximum pore diameter in (μm).

γ = surface tension of test liquid in (dynes/cm) at the temperature of the test, and for water = 72 (dyne/cm) at 20°C

P_r = pressure, (mm Hg).

Also we can measure the max. pore diameter theoretically by the relationship with (K), where K in (cm^2) is related to a mean (or "effective") pore diameter $D_{\max.}$ (in micron) by:

$$K = 0.617 \cdot 10^{-11} \cdot D_{\max.}^2 \dots\dots\dots (8-2)$$

$K = 1 \text{ cm}^2/\text{sec}$ is equivalent to $K = 1.02 \cdot 10^{-5} \text{ cm}^2$. ⁽⁷⁴⁾

2-13-2 Mechanical properties:-

1-compressive strength:-

An important consequence of the theory that brittle fracture is initiated at Griffith flaws is that the fracture strength of brittle solids is statistical in nature, depending on the probability that a flaw capable of initiating fracture at a specific applied stress is present. This is the main explanation of the scatter of results normally found for the strength of ceramic materials. ⁽⁷⁷⁾

$$\bar{\sigma}_f = k d^{-1/2}$$

Where:

$\bar{\sigma}_f$: fracture strength.

k: constant depending on the material properties.

d: grain size diameter.

This equation is known as the "Orowan" relation, where the sizes of the initial flaws are limited by the grains and scale with the grain size and brittle behavior is observed. ⁽⁷⁷⁾

Compressive strength is calculated from the equation below:

$$\bar{\sigma}_c = F/A_r \dots\dots\dots (9-2)$$

Where:

$\bar{\sigma}_c$: compressive strength in (MPa).

F: load applied until fracture (N).

A_r : cross section area (mm²).

2- Hardness:-

Filter hardness represent the filter ability to resist the penetration and deformation by other materials during filtration process.

Usually filter hardness measured according with ASTM D217-97; the test consists of measuring the amount of penetration into the filter achieved by a weight cone under certain specific condition. ⁽⁷⁸⁾

Because this apparatus is not available we measure Brinels hardness for samples.

2-14 Dyes concentration determination:-

A useful analytical tool for determining the concentration of colored material in solution is absorbance spectrophotometer. Colored substances absorb light in the visible electromagnetic spectrum. The amount of light absorbed by a substance in solution is easily measured by a photocell detector and is proportional to the solute concentration.

If I_0 is the intensity of light entering a solution and I_t is the intensity of light exiting the solution, then the transmittance, T, of the solution is

given as I_t/I_0 . Transmittance is also expressed as a percentage, (I_t/I_0) (100%). Because it relates more directly to solute concentration, the absorbance A is more often used to express the amount of light a solution absorbs. $Abs. = -\log (T)$ or $Abs. = \log (I_t/I_0)$. At a given wavelength of light, absorbance depends on the absorptivity of the solute, the length of solution the light passes through and the solute concentration. This relationship, known as Beer's law, is expressed as

$$Abs. = \alpha * C * L \dots \dots \dots (10-2)$$

Where:

Abs. = absorbency (%).

C = concentration of the sample (ppm).

L= the path length of the cell (mm).

α = the extinction coefficient (constant).

Here “ α ” is a proportionality constant (molar absorptivity for molar concentration units), “L” is the path length through the solution, and “C” is the solute molar concentration, and we can either measure %T and convert it to Abs. values or to measure absorbance directly from the spectrophotometer.

In Beer’s Law experiments, it is necessary to find a wavelength at which light is absorbed strongly by the solute being studied, without interference by other absorbing solutes and where the absorbance does not change rapidly with changing wavelength. Then it is necessary to measure the absorbance of several solutions of known concentration. So a calibration curve can be drawn. ⁽⁷⁹⁾

Concentration of dyes usually measured by using UV-spectrophotometer that measure dye absorbency and according to Beer’s law, Absorbance is directly proportional to concentration: ⁽⁸⁰⁾

Procedure: In order to use the UV-Visible spectrophotometer the wavelength of light that is to be emitted onto the sample must be first determined. This is done by first setting the wavelength at a value between 280 to 600 nm. Two cells were rinsed with distilled water, and the absorbance was adjusted to zero. The reference cell was left in the machine. The sample cell was emptied and filled with the stock (100%) solution of dye. The spectrum was scanned between 280 and 600 nm, and the wavelength at which absorbance is maximum was determined. The spectrophotometer was then set to this wavelength and the absorbances of the solutions were measured for all dye samples. ⁽⁸⁰⁾

2-15 Filter efficiency:-

The efficiency of dye reduction by filters can be calculated from the initial and final dye concentration, where initial concentration is known to us and final concentration can be calculated from Beer's law directly.

Efficiency of filters expresses as percentage and calculated from the equation:

$$\epsilon\% = \{(C_o - C_f)/C_o\} * 100 \dots \dots \dots (11-2)$$

Where:

ϵ : Filter efficiency (%).

C_o : Initial dye concentration (ppm).

C_f : Final dye concentration (ppm).

Chapter Three

EXPERIMENTAL WORK

3-1 Introduction

This study includes the process of making SBACF from (AC) and local raw material (HDPE) by dry, single direction pressing and then making tests on the produced samples which include mechanical, physical and chemical tests.

The process for producing SBACF from local raw materials (AC and HDPE) includes the following stages:

- 1- Preparation of raw materials.
- 2- Samples formation.
- 3- Finishing of samples.
- 4- Testing of samples.

All the above steps are shown in Fig. 3-1.

3-2 Raw materials

Two materials are used to produce SBACF which are:

- a) Activated carbon: - Activated carbon used in this study has the specification shown in the Table 3-1; It represents the matrix material in the body of the filter.

Three sizes of carbon particle were used in the process for making the samples and which will be explained later.

- b) High density polyethylene (HDPE): - Is used as binder material in the process to adhere the carbon particles with each other. HDPE used in this study is produced in the general company for petrochemical industries -Basrah- Iraq, and have the specification listed in Table 3-2.

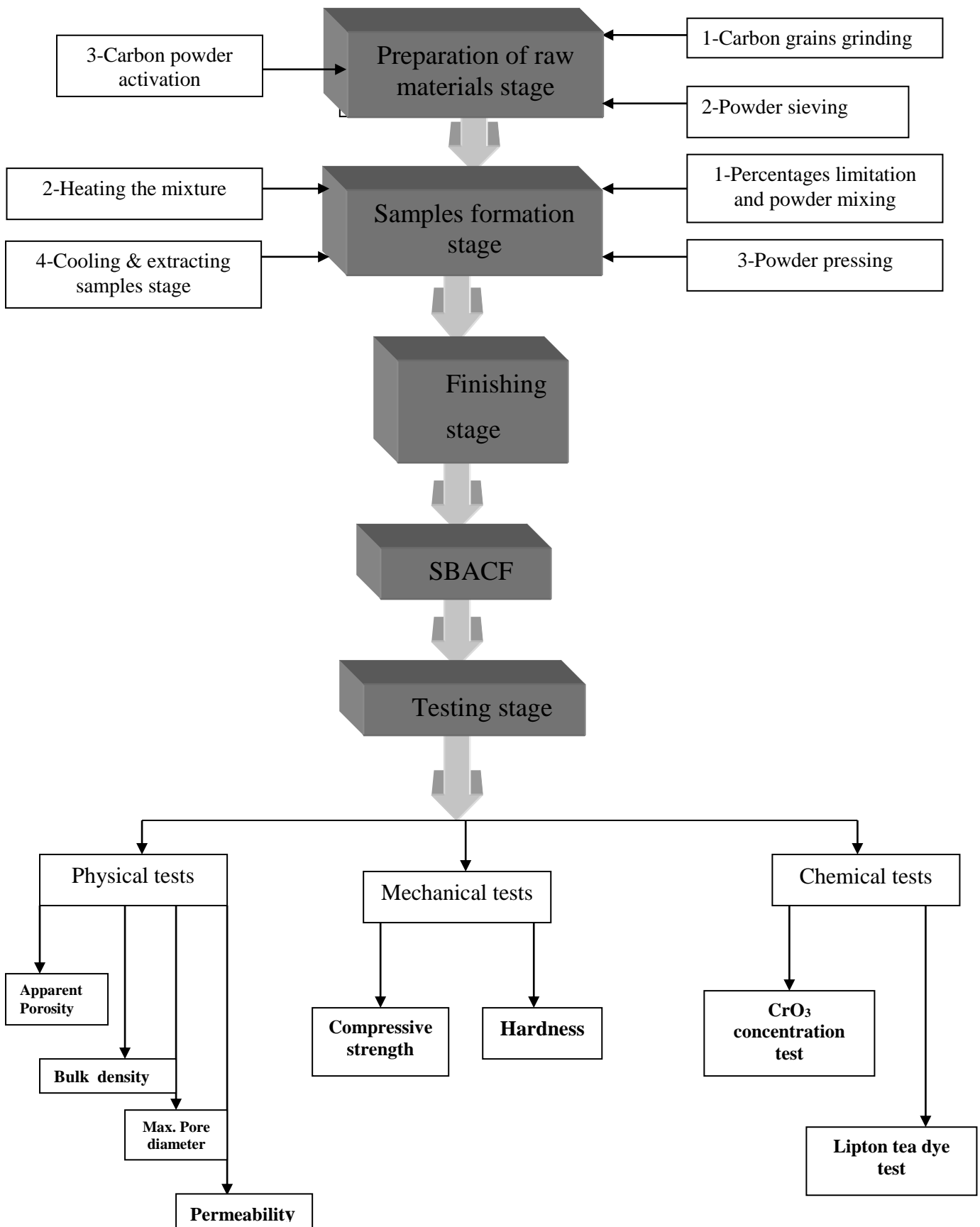


Figure 3-1: Experimental flow chart.

Table 3-1:- Properties of Activated carbon used.

<i>Test</i>	<i>Specification</i>
<i>Dimension (granular)</i>	<i>12x40 mesh (0.4-1.6)mm</i>
<i>Bulk density (kg/m³)</i>	<i>460-480</i>
<i>Void fraction</i>	<i>0.45</i>
<i>Specific surface area (m²/g)</i>	<i>1100-1130</i>
<i>Ash (%)</i>	<i>5 (max.)</i>
<i>Microporos</i>	<i>high</i>

Table 3-2:- Properties of HDPE used.

<i>Source</i>	<i>general company for petrochemical industries - Basrah- Iraq-</i>
<i>class</i>	<i>6030</i>
<i>Melt index</i>	<i>(2.5-4) g/10 min.</i>
<i>density</i>	<i>0.960 g/ cm³</i>

3-3 Preparation of raw materials:

This stage includes raw materials preparation that used in making SBACF which are (AC and HDPE). The explanation of this process is as follows:-

1- Carbon grinding: Activated carbon used in this study had large grains which have to be ground mechanically in the laboratory to get particles as small as (<150µm).

2- Carbon powder sieving: - Three carbon particle sizes were used in this study. These particle sizes are got by sieving method

using three different sieves. The selected sizes in this study are explained in Table 3-3, while the size of binder material powder (HDPE) is less than (75 μm) for all mixtures.

3- Carbon powder reactivation: - After grinding and sieving stage we put carbon powder in the oven at (150 °C) for (1 hour). The purpose of this stage is to burn out the organic material if it is existed. In addition to that the reactivity of carbon powder will be gained which is affected by mechanical work (crushing and sieving).

Table 3-3:- The selected carbon particle size. (ASTM Standards)

Mesh	<i>opening size (μm)</i>
100	150
150	90
200	75

3-4 Samples formation stage

This stage includes the following steps respectively:

1- Percentages limitation and powder mixing: - this stage consists of two steps:

a) **Percentages limitation:** includes choosing of binder material percents which will be mixed with activated carbon powder.

Three percents of binder material have been tested (10, 20, and 30) % from each sample weight and the rest of the sample weight was activated carbon powder.

b) **Mixing process:** this process accomplished by using electrical mixer where each batch spends three hours in the mixer to get the final batch

that will be used to manufacture SBACF, Table 3-4 represents the mixing percentages which were used in SBACF forming.

2- Heating the mixture: - After finishing the batch preparation the limited quantity of powder was put in the die to form the sample according to desired test type. Every test has different sample shape. After this we put the die filled with powder in the oven at 200 °C. The purpose of this

Table 3-4:- Mixing percents that used in SBACF forming.

<i>No.</i>	<i>AC %</i>	<i>HDPE %</i>	<i>HDPE Particle size(μm)</i>	<i>AC Particle size(μm)</i>
1	90	10	<75	75
2	90	10	<75	90
3	90	10	<75	150
4	80	20	<75	75
5	80	20	<75	90
6	80	20	<75	150
7	70	30	<75	75
8	70	30	<75	90
9	70	30	<75	150

process is to make the binding material (HDPE) to become tacky but not melted to bind the carbon particles together without excessively wetting the carbon particles when melted. In this way the surface area of the carbon particles will be better occluded.⁽⁸¹⁾

The period of soaking time in the oven ranging from (7-10) min. according to powder (AC+HDPE) quantity in the die, the period decreases with decreasing the powder quantity.

3- Powder pressing: - Single direction dry pressing method was used in sample pressing by using hydraulic press as shown in Fig. 3-2.

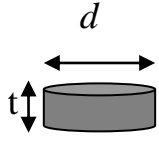
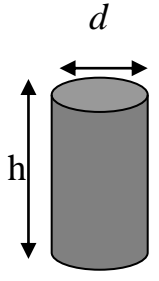
Sample weight, shape and their dimensions depend on the property of sample wanted to study as shown in the Table 3-5. Two shapes of steel dies adapted according to ASTM standards were used to form all samples while the load values were (10, 20 and 30) tons, the rate of pressing is constant for all samples which was (2.5 ton/min.).

4- Cooling & extracting samples: - After reaching the specific pressure in pressing stage the load remains applied on the die until the die cools to room temperature. After that the load will be removed and sample will be extracted from the die.



Fig. 3-2:- The hydraulic press.

Table 3-5: Die shapes and samples weight.

NO.	Property	Die dimensions(mm)	Sample dimensions(mm)	Sample shape	sample weight(g)
a	Physical properties and hardness	$D_o=50$ $D_{in}=32$ $H=60$	$d=32$ $t=8$		5
b	mechanical properties (compressive strength)	$D_o=15$ $D_{in}=10$ $H=60$	$d=10$ $h=20$		3

3-5 Finishing stage:

In this stage increments are removed from the edges of the samples and are prepared for testing. Also the die is cleaned and oiled for the next pressing process.

3-6 Testing stage:

Many tests have been accomplished on the produced samples in order to study the properties of carbon filter. These tests can be classified into three types:-

3-6-1 Physical Tests:- many physical tests were performed on the samples which are:

3-6-1-1 Apparent porosity: -

In these tests we depend on the ASTM standard (C-373-88) and each test result is the average of three samples as shown in the following steps.⁽⁸²⁾

1- Drying the test specimens to constant mass by heating in an oven at 105°C, followed by cooling in a desiccator. The dry mass (D), was determined to the nearest 0.01 g.

2- Immersing the specimens in a pan of distilled water and boiling them for 5 h, taking care that the specimens are covered with water at all times. Using setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. After 5 h boiling, allowing the specimens to soak for an additional 24 h.

3- After impregnation of the test specimen in water, then the suspended mass (S), was determined to the nearest 0.01 g, while suspended in water.

4- After determination of the suspended mass, each specimen was blotted lightly with a moistened cotton cloth to remove all excess water from the surface. The saturated mass (M), was determine to the nearest 0.01 g after rolling the specimen lightly on the wet cloth.

Excessive blotting of specimen will introduce error by withdrawing water from the pores of the specimen. Make the weighing immediately after blotting, the whole operation being completed as quickly as possible to minimize errors caused by evaporation of water from the specimen.

3-6-1-2 Bulk Density:-

Density of samples had been measured according to ASTM standard (C373-88) as mentioned before in chapter two. Each test result represents the average of three samples.⁽⁸²⁾

The bulk density of samples was calculated from equation (4) presented in chapter two.

3-6-1-3 Permeability:-

Permeability of filter samples had been measured according to Darcy's law as explained in chapter two. The apparatus used in measuring the permeability is shown in figure 3-3 which is locally built.

The method of measuring samples permeability is as following:-

- 1- Put the sample at the base of the apparatus and sealed it by a rubber ring to ensure that water has no way out through the sides of sample but through the center.
- 2- After that fill the apparatus tube with distilled water at room temperature.
- 3- Applying air pressure at (0.6 bar) for 10 min. and collecting the water that passes through the sample in a volumetric beaker.

Repeating the above steps for all samples and each result represents the average of three samples.

Permeability was calculated by using Darcy's law directly.

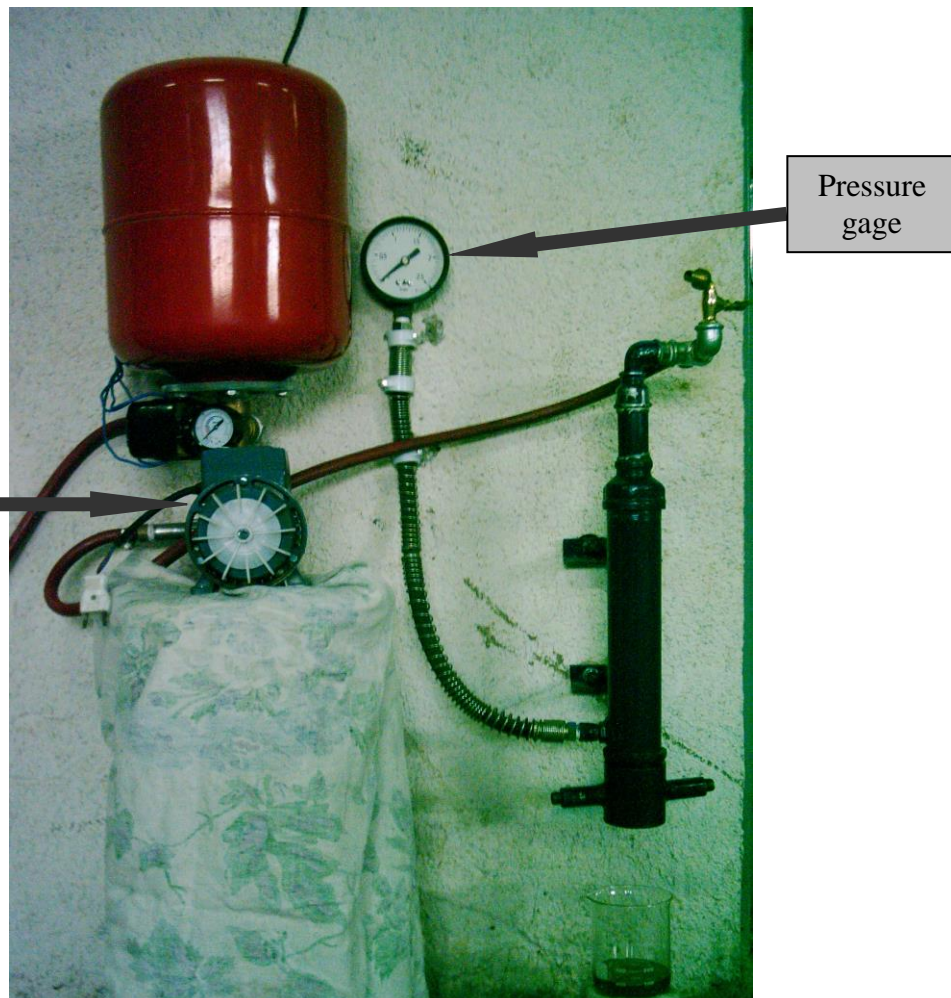




Fig. 3-3: Permeability measuring apparatus.

3-6-1-4 Maximum Pore Diameter: -

Maximum pore diameter in the carbon filter sample has been calculated by using apparatus shown in Fig. 3-4 which is locally built. Experimental max. pore diameter calculated according to ASTM standard (E 128-99) as explained in chapter two.⁽⁷⁶⁾

Using equation (7-2) for experimental max. pore diameter and equation (8-2) for theoretical max. pore diameter in samples as explained before in chapter two.

Each test result represents the average of three samples.

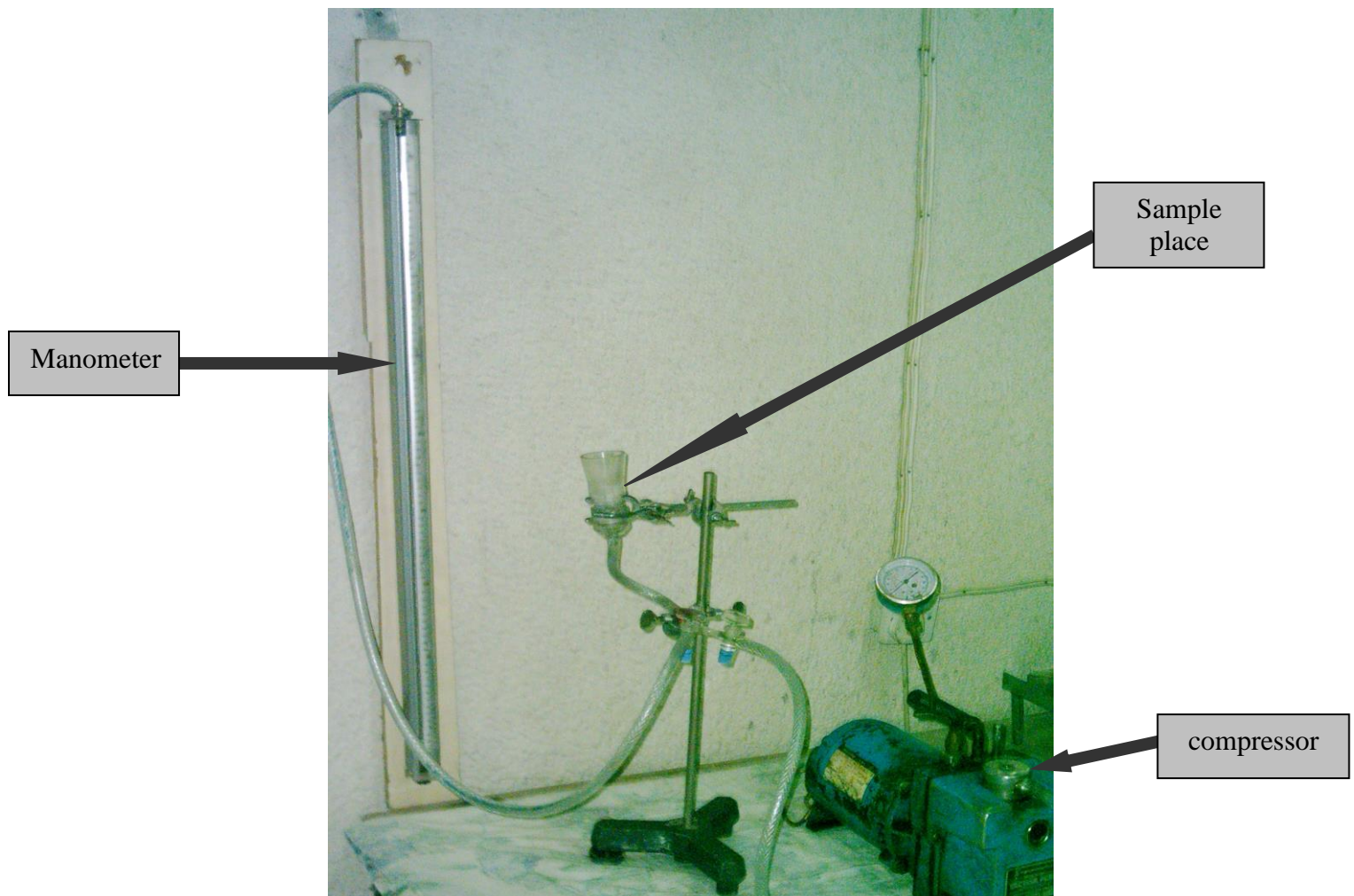


Fig 3-4: Max. pore diameter measuring apparatus.

3-6-2 Mechanical Tests:- Material strength in ceramic is the material resistance to fracture when load applied on it.

3-6-2-1 *Compressive strength:-*

Most ceramic materials have high compressive strength; sample for this test could be rectangular, cubic or cylinder. When we do this test for ceramic bodies, the length of the sample should be twice it is diameter ($h= 2d$) and the surfaces of the samples should be even and flat. Compressive strength measured using general testing machine shown in figure 3-5. Each test result is the average of three test samples. This test is done according to the ASTM (C 773-88) standard as explained in chapter two. ⁽⁸³⁾



Fig 3-5: general testing machine

3-6-2-2 Hardness test: - Brinell hardness test have been made for all samples by using small 2.5 mm diameter steel ball and (187.5 g) load applied for (3 min.). The results were taken directly from the Brooks Inspection hardness tester apparatus.

3-6-3 Chemical Tests: -

Two types of chemical tests have been made, Lipton tea dye concentration test and CrO_3 concentration test. Their spectra are shown in figures 3-6 & 3-7 respectively. Using UV-Visible Spectrophotometer shown in figure 3-8, we consider tea dye as an organic contaminant in water that contains caffeine matter, while chromium oxide will be considered as inorganic contaminant and heavy metal common in wastewater. Preparation of dyes and way of testing are explain below:

A) *Tea dye:* -

Tea dye was prepared by using Lipton tea bags that weight approximately (2g). Each bag was immersed in (100 ml) of boiling distilled water for (2min.). To let the stock dye get the original concentration which passes through all filter samples. We do the test by filling (200 ml) of stock dye ,with initial concentration of ($C^{\circ}=20000\text{ppm}$), in the permeability apparatus and then applying (0.6 bar) air pressure to force the dye to penetrate the filter sample. Then we collect the permeable dye in a beaker for testing by spectrophotometer,

and the way of choosing the maximum absorbance value and setting the spectrophotometer had explained in chapter two.

In figure 3-6 there was a peak at 340nm represents the maximum absorbency value and it was chosen for tea dye samples testing.

Dye absorbency and concentration were calculated directly by using Beers law as explained in chapter two.

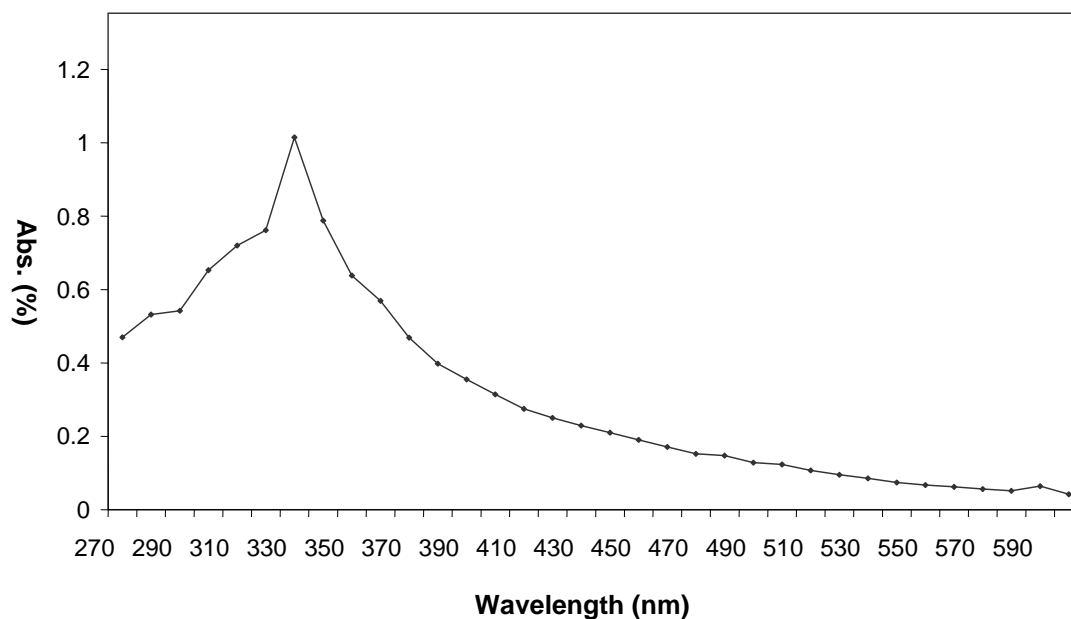


Fig 3-6: Absorption curve for tea dye ((C=50% C_o) =10000ppm)

The measured value of pH of tea dye was 5.24 at room temperature.

B) CrO₃:-

Chromium oxide is considered as a heavy metal and prepared by placing (0.1 g) from CrO₃ powder in (100 ml) distilled water at room temperature to get the stock dye with concentration (1000 ppm) which is penetrating through all filter samples, with the same procedure carried out for CrO₃, with the peak occurring at 460nm and it was chosen for CrO₃ samples testing.

Dye absorbency and concentration were calculated directly by using Beers law as explained in chapter two.

Samples efficiency for both dies was calculated as mentioned before in chapter two.

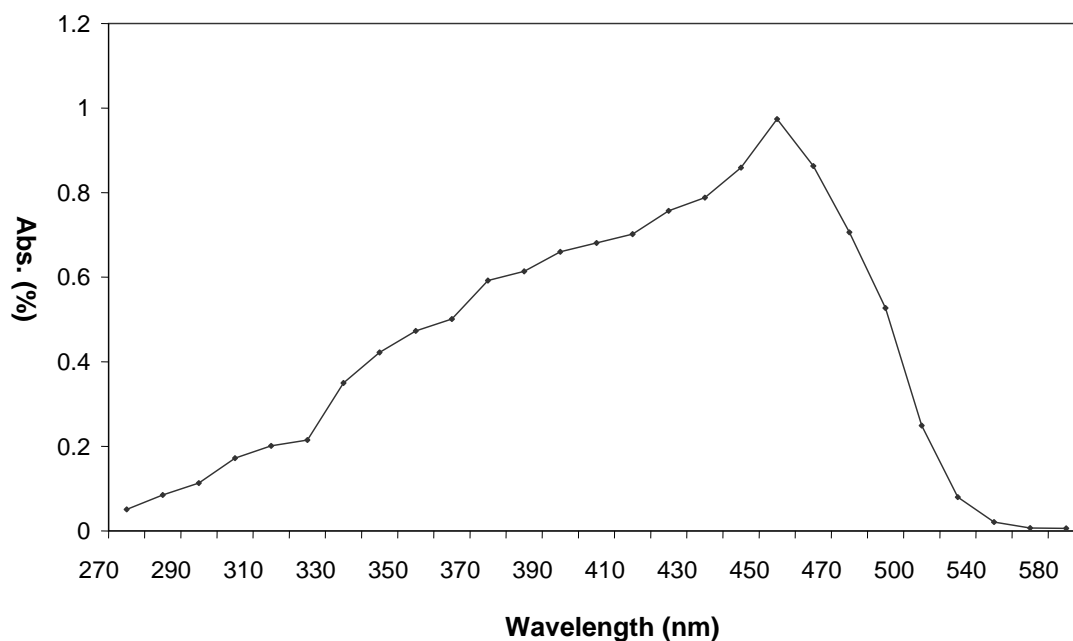


Fig 3-7: Absorption curve for CrO₃ ((C=50% C_o) =500 ppm)

The pH value of CrO₃ dye was 1.82 when measured at room temperature using pH meter.



Fig. 3-8: UV-Visible Spectrophotometer.

Chapter Four

RESULTS AND DISCUSSION

4-1 Introduction

In this chapter the results obtained from experiment tests are discussed, which describe the carbon filter properties. Also it discusses the effect of the following factors (carbon particle size, binder percent and the compaction pressure) on the filter properties to specify the best characteristics for the filter.

4-2 physical properties

4-2-1 Apparent Porosity: -

Fig. 4-1 show the effect of carbon particle size on apparent porosity of the filter samples with different percentage of binder material and compaction pressure. The maximum values for it are at largest carbon particle size. We think that it is because at large particle size the number of particles in unit volume will be less than that at fine particles size which leaves large pores, and increase porosity. Also the particle size distribution may appreciably affect the resulting porosity, as small particles may occupy pores formed between the large particles, thus reducing porosity. Other factor that effect porosity is the compaction pressure that decreases these properties by increasing compaction pressure as can be seen from Fig. 4-1.

The effect of binder percent on apparent porosity of the filter samples is decreasing it with increasing binder percent, the reason of that is back to that the increasing in binder percent will give the samples very good compaction and make particles close to each other also clog the pores in the body as it can be seen from Fig. 4-1.

4-2-2 Bulk Density: -

Fig. 4-2 shows the effect of carbon particle size on bulk density of the filter samples with different percentage of binder material and compaction pressure, where density of the body is increasing in general with increasing the following: ⁽⁸⁴⁾

- 1- The range of particle sizes.
- 2- The compaction pressure used in the compaction process.
- 3- Decreasing the applied force rate by compaction.
- 4- And with decreasing particle hardness.

Fig 4-2 also shows the effect of binder percentage on density of filter samples. The density is increasing with increasing binder percentage. That can be attributed to the density of binder material which is higher than that of the carbon powder. This coincides with the porosity where the increase in porosity will lead to decrease the density because we get a large volume at constant mass. ⁽⁸⁵⁾

4-2-3 Max. Pore diameter: -

Fig. 4-3 shows the effect of carbon particle size on max. pore diameter of the filter samples with different percentage of binder material and compaction pressure. The increase in particle size will increase the max. pore diameter. The reason of that is due to particles arrangement with each other. The large particles will form larger pores than that fine particles do.

Fig. 4-3 also shows the effect of binder percentage on the Max. pore diameter of the filter samples where the increase in binder percent will decrease the max. pore diameter existing in the sample. We expect that the increasing in binder percent will give good compaction in samples. Also the binder materials will form shells around the particles which fill the pores and minimize their sizes.

The increase in compaction pressure will decrease the max. Pore diameter because higher value of compaction pressure will give a coherent body and better arrangement for the particles to fill large pores.

The discussion above also agrees with the theoretical calculations for the max. Pore diameter which were explained in chapter two and as shown in Fig. 4-4.

4-2-4 Permeability: -

Fig. 4-5 shows the effect of carbon particle size on the permeability of the filter samples with different percentage of binder material and compaction pressure. According to the Darcy's law the permeability increases with the increase of pore diameter which increases with increasing particle size.

The permeability of the body is connected directly to the number of the open channels that connect pores and the exterior wall of the body. The increase in binder percentage decreases the permeability through the decrease in pore diameter and number of pores as shown in Fig. 4-5.

Compaction pressure effects inversely on the permeability where increasing in the pressure value decrease the permeability of filter samples. That's due to the increase in compaction pressure will decrease the pore diameter which decrease the filters volumetric flow.

4-3 Mechanical Tests

4-3-1 Compressive strength: -

Fig. 4-6 shows the effect of carbon particle size on the compressive strength of the filter samples with different percentage of binder material and compaction pressure. The maximum value for the compressive strength is at minimum carbon particle size and decreases with increasing particle size. That can be explained on the base that the strength vary with grain size inversely as mentioned in chapter two.

Fig. 4-6 also shows the effect of binder percentage on the compressive strength of the filter samples. The increasing in binder percent increases the strength that's because the increasing in binder percentage will increase carbon particles adhesion, thereby the strength of the body will be improved. Also the increasing in compaction pressure will increase the strength that's because of the minimizing the porosity (voids) of the body which will be considered as weak points in the body which responsible for beginning of fracture (crack growth).⁽⁸⁶⁾

4-3-2 Hardness: -

Hardness of samples is affected by the three variables (Carbon particle size, binder percentage and compaction pressure) as in compressive strength. Hardness is decreasing slightly with the increase of particle size and increasing with the increase of binder percentage and compaction pressure as shown in Fig. 4-7.

4-4 Chemical Tests

Tea dye and CrO₃ concentration determination: -

Figs. 4-8 and 4-9 show the effect of carbon particle size on the absorption process for tea dye and CrO₃ respectively with different percentage of binder material and compaction pressure. The decrease in carbon particle size decreases the absorption values as explained in chapter two. The major factor in decreasing the concentration of both contaminants is the efficiency of adsorption process, where adsorption process is affected by many factor as explained in chapter two.

The effect of binder percentage on the absorption process for tea dye and CrO₃ is decreasing in absorption values when binder percentage is decreasing and that's leads to decrease the dye and oxide concentration in water. The reason of that is due to the increments in binder percentage that reduces the quantity of carbon powder which is responsible for the adsorption process also it reduces the number of pores (porosity) and there volume and make shells around carbon particles which hinder the contaminants molecules to reach the pores where adsorption process happens.

The flow conditions of the binder during processing are critical for obtaining the proper balance between strength and adsorption. If a resin has low flow characteristics, then it does not coat the carbon particles sufficiently to bond them together effectively. If a resin displays high flow characteristics, then it tend to fill up the microporosity in the carbon thereby taking away adsorption sites that are needed to achieve high adsorption capacity in the body, the resin must be able to flow sufficiently to bind the

carbon particles together thus contributing strength to the body but not to block the porosity. ⁽⁸⁷⁾

The increase in compaction pressure will decrease the absorption values. We think that the increase in compaction pressure reduces porosity in the same time it minimize pores diameters which hinder dies molecules to pass through and to be adsorb.

Figs. 4-10 and 4-11 show the effect of carbon particle size on concentrations of tea dye and CrO_3 in water respectively with different percentage of binder material and compaction pressure. The decrease in carbon particle size will decrease the dye and oxide concentration after filtration. That can be understood from Beer's law where dye concentration is proportional directly with absorbency as mentioned in chapter two.

Carbon block filter manufacture can be complicated and typically involves a careful balance of various competing interests. It has been known that smaller carbon particles provide improved filtration. This is due in large part to the fact that smaller particles provide increased surface area and create a denser block that traps smaller particulate matter. Unfortunately smaller carbon particle sizes create several problems. First, water flow rates through the filter are dramatically affected, sufficiently reducing the amount of water that can be treated in a given amount of time. Second, a high percentage of smaller carbon particles make it difficult to manufacture the carbon block using conventional manufacturing techniques. More specifically, higher levels of fine particles interfere with the binder's ability to make a solid carbon block. This causing cracking, crumbling and other defects in the carbon block. ⁽⁸⁸⁾

Figs. 4-12 and 4-13 show the effect of carbon particle size on the filter efficiency for removing tea dye and CrO_3 respectively with different percentage of binder material and compaction pressure. Efficiency of filter samples increase by increasing the reduction of dyes concentration as can be understood from the efficiency equation mentioned in chapter two. Filters made out of fine particles are most effective than that of coarse particles. As we mentioned before, this is due to the fine particles have large inner surface area.

The effect of increasing binder percentage is a reduction in filtration efficiency, because the carbon surface is reduced which is responsible for the adsorption of the dyes.

Chapter Five

CONCLUSIONS AND RECOMMENDATIONS

5-1 Conclusions

According to data of the present study, the following conclusions are obtained:-

- 1- Porosity of filter samples is increased with increasing carbon particle size and decrease with increasing the binder percent and the compaction pressure.
- 2- Density of filter samples is increased with increasing compaction pressure and binder percentage and decreased with increasing carbon particle size.
- 3- Permeability of filter samples increases with increasing the carbon particle size and decreases with increasing the binder percentage and the compaction pressure.
- 4- In opposite to permeability, both compressive strength and hardness of filter samples increase with increasing the binder percentage and compaction pressure and decrease with increasing carbon particle size.
- 5- Tea dye removal efficiency of filter samples reached 99.9%, where CrO_3 removal from water reached 99.5%.
- 6- The efficiency of adsorption process increases with decreasing carbon particle size, binder percentage and the compaction pressure.

5-2 Recommendations for future work

- The following recommendations can be helpful for further study:-
- 1- This work can be extended to study the effect of pH, temperature, flow rate and concentration of the dies on the efficiency of the same filter samples.
 - 2- Testing other water contaminants such as another heavy metal, taste, odor and microorganism contaminants using the same treatment process.
 - 3- Study the system pilot scale and scaling up the designed data for filter units.
 - 4- Add other additives to the original raw materials mixture such as brass and silver to prevent the bacteria growth problem.
 - 5- Using another thermoplastic binder and comparing the result with this study.
 - 6- Using a blend from carbon particle sizes instead of single particle size in each filter sample.

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APPENDIX-A

Apparatus and Equipment used in this study:

<i>APPARATUS AND EQUIPMENT</i>	<i>SPECIFICATIONS</i>
<i>sensitive balance</i>	<i>made in Germany, type (Startorius)</i>
<i>Electrical mixer</i>	-
<i>Sieves</i>	<i>Wykeham Farrance, engineering Ltd., slough_England</i>
<i>electrical oven</i>	<i>Prodit _ Italy, Santena (Torino)</i>
<i>Dies</i>	<i>from medium carbon steel</i>
<i>Hydraulic press</i>	<i>Soiltest, INC. Evanstone, Illinois, USA</i>
<i>General testing machine</i>	<i>Phywe/ Germany, equipment system for strain Gauge measuring techniques.17596.93/Nr.519</i>

APPENDIX-A

<i>Mercury Manometer</i>	<i>made in USA</i>
<i>Beakers and Funnels</i>	<i>resistance to thermal shock</i>
<i>Timer</i>	<i>water resistance, made in Japan</i>
<i>Vacuum motor</i>	<i>thermally protected, General electric A. C. motor (55KH35KG1363T), USA.</i>
<i>permeability measuring apparatus</i>	-
<i>Brooks Inspection Equipment Ltd.</i>	<i>Colchester, England</i>
<i>UV-Visible Spectrophotometer- Single Beam</i>	<i>Sheizieno- Japan</i>
<i>Digital pH meter</i>	<i>Hanaa, Roman</i>

Appendix -B

Each sample carried three digits read it from right to left, first digit represents the particle size and as follow:

Digit	Represents
1	75 μm
2	90 μm
3	150 μm

Second digit represents the binder material percentage and as follow:

Digit	Represents
1	10 %
2	20 %
3	30 %

Third digit represents the compaction pressure and as follow:

Digit	Represents
1	10 Ton
2	20 Ton
3	30 Ton

Appendix -B

COMPRESSIVE STRENGTH (MPa)		10 TON
Particle size	$\bar{\sigma}$(MPa)	symbol
75	4.586	111
90	4.076	112
150	3.439	113
75	8.662	121
90	7.77	122
150	6.751	123
75	27.133	131
90	22.42	132
150	12.738	133

COMPRESSIVE STRENGTH (MPa)		10 TON
Binder %	$\bar{\sigma}$(MPa)	symbol
10	4.586	111
20	8.662	121
30	27.133	131
10	4.076	112
20	7.77	122
30	22.42	132
10	3.439	113
20	6.751	123
30	12.738	133

COMPRESSIVE STRENGTH (MPa)		20 TON
Particle size	$\bar{\sigma}$(MPa)	symbol
75	6.751	211
90	5.095	212
150	4.84	213
75	9.044	221
90	8.28	222
150	7.643	223
75	31.974	231
90	28.407	232
150	24.458	233

COMPRESSIVE STRENGTH (MPa)		20 TON
Binder %	$\bar{\sigma}$(MPa)	symbol
10	6.751	211
20	9.044	221
30	31.974	231
10	5.095	212
20	8.28	222
30	28.407	232
10	4.84	213
20	7.643	223
30	24.458	233

COMPRESSIVE STRENGTH (MPa)		30 TON
Particle size	$\bar{\sigma}$(MPa)	symbol
75	7.102	311
90	5.681	312
150	5.034	313
75	9.520	321
90	8.603	322
150	8.820	323
75	35.137	331
90	31.725	332
150	27.911	333

COMPRESSIVE STRENGTH (MPa)		30 TON
Binder %	$\bar{\sigma}$(MPa)	symbol
10	7.102	311
20	9.520	321
30	35.137	331
10	5.681	312
20	8.603	322
30	31.725	332
10	5.034	313
20	8.820	323
30	27.911	333

Appendix -B

PORE DIAMETER (μm)		10 TON
Binder %	Exper.	symbol
10	5.204	111
20	3.428	121
30	3.085	131
10	6.923	112
20	4.547	122
30	3.789	132
10	8.307	113
20	6	123
30	4.988	133

PORE DIAMETER (μm)		10 TON
Binder %	Theo.	symbol
10	4.425	111
20	3.633	121
30	2.2	131
10	4.706	112
20	4.287	122
30	2.287	132
10	4.896	113
20	4.441	123
30	2.357	133

PORE DIAMETER (μm)		20 TON
Binder %	Exper.	symbol
10	4.5	211
20	3.223	221
30	3.085	231
10	6.171	212
20	4.21	222
30	3.483	232
10	7.448	213
20	5.61	223
30	4.5	233

PORE DIAMETER (μm)		20 TON
Binder %	Theo.	symbol
10	4.307	211
20	3.38	221
30	1.485	231
10	4.342	212
20	4.166	222
30	1.642	232
10	4.576	213
20	4.254	223
30	1.663	233

PORE DIAMETER (μm)		30 TON
Binder %	Exper.	symbol
10	4.302	311
20	3.017	321
30	3.015	331
10	5.896	312
20	4.022	322
30	3.17	332
10	7.205	313
20	4.866	323
30	4.227	333

PORE DIAMETER (μm)		30 TON
Binder %	Theo.	symbol
10	4.195	311
20	3.303	321
30	1.280	331
10	4.294	312
20	3.951	322
30	1.442	332
10	4.365	313
20	3.996	323
30	1.557	333

Appendix -B

PORE DIAMETER (μm)		10 TON
Particle size	Exper.	symbol
75	5.204	111
90	6.923	112
150	8.307	113
75	3.428	121
90	4.547	122
150	6	123
75	3.085	131
90	3.789	132
150	4.988	133

PORE DIAMETER (μm)		10 TON
Particle size	Theo.	symbol
75	4.425	111
90	4.706	112
150	4.896	113
75	3.633	121
90	4.287	122
150	4.441	123
75	2.2	131
90	2.287	132
150	2.357	133

PORE DIAMETER (μm)		20 TON
Particle size	Exper.	symbol
75	4.5	211
90	6.171	212
150	7.448	213
75	3.223	221
90	4.21	222
150	5.61	223
75	3.085	231
90	3.483	232
150	4.5	233

PORE DIAMETER (μm)		20 TON
Particle size	Theo.	symbol
75	4.307	211
90	4.342	212
150	4.576	213
75	3.38	221
90	4.166	222
150	4.254	223
75	1.485	231
90	1.642	232
150	1.663	233

PORE DIAMETER (μm)		30 TON
Particle size	Exper.	symbol
75	4.302	311
90	5.896	312
150	7.205	313
75	3.017	321
90	4.022	322
150	4.866	323
75	3.015	331
90	3.17	332
150	4.227	333

PORE DIAMETER (μm)		30 TON
Particle size	Theo.	symbol
75	4.195	311
90	4.294	312
150	4.365	313
75	3.303	321
90	3.951	322
150	3.996	323
75	1.280	331
90	1.442	332
150	1.557	333

Appendix -B

PERMEABILITY (mm darcy)		10 TON
Particle size	K	symbol
75	12.245	111
90	13.85	112
150	14.986	113
75	9.986	121
90	11.492	122
150	12.334	123
75	3.003	131
90	3.27	132
150	3.474	133

PERMEABILITY (mm darcy)		10 TON
Binder %	K	symbol
10	12.245	111
20	9.986	121
30	3.003	131
10	13.85	112
20	11.492	122
30	3.27	132
10	14.986	113
20	12.334	123
30	3.474	133

PERMEABILITY (mm darcy)		20 TON
Particle size	K	symbol
75	11.597	211
90	11.788	212
150	13.091	213
75	8.254	221
90	10.851	222
150	11.317	223
75	1.38	231
90	1.686	232
150	1.73	233

PERMEABILITY (mm darcy)		20 TON
Binder %	K	symbol
10	11.597	211
20	8.254	221
30	1.38	231
10	11.788	212
20	10.851	222
30	1.686	232
10	13.091	213
20	11.317	223
30	1.73	233

PERMEABILITY (mm darcy)		30 TON
Particle size	K	symbol
75	11.002	311
90	11.531	312
150	11.913	313
75	7.143	321
90	10.16	322
150	10.4	323
75	1.025	331
90	1.301	332
150	1.516	333

PERMEABILITY (mm darcy)		30 TON
Binder %	K	symbol
10	11.002	311
20	7.143	321
30	1.025	331
10	11.531	312
20	10.16	322
30	1.301	332
10	11.913	313
20	10.4	323
30	1.516	333

Appendix -B

APPARENT POROSITY		10 TON
Particle size	%P	symbol
75	52.4204	111
90	58.259	112
150	60.824	113
75	42.0905	121
90	57.931	122
150	58.840	123
75	31.3139	131
90	50.076	132
150	51.1146	133

APPARENT POROSITY		10 TON
Binder %	%P	symbol
10	52.4204	111
20	42.0905	121
30	31.3139	131
10	58.259	112
20	57.931	122
30	50.076	132
10	60.824	113
20	58.840	123
30	51.1146	133

APPARENT POROSITY		20 TON
Particle size	%P	symbol
75	48.9634	211
90	58.9	212
150	59.593	213
75	36.5371	221
90	55.405	222
150	57.579	223
75	29.5202	231
90	47.855	232
150	51.114	233

APPARENT POROSITY		20 TON
Binder %	%P	symbol
10	48.9634	211
20	36.5371	221
30	29.5202	231
10	57.3	212
20	55.405	222
30	47.855	232
10	59.593	213
20	57.579	223
30	51.114	233

APPARENT POROSITY		30 TON
Particle size	%P	symbol
75	44.634	311
90	48.259	312
150	55.630	313
75	31.702	321
90	47.655	322
150	52.840	323
75	27.831	331
90	43.891	332
150	46.621	333

APPARENT POROSITY		30 TON
Binder %	%P	symbol
10	44.634	311
20	31.702	321
30	27.831	331
10	48.259	312
20	47.655	322
30	43.891	332
10	55.630	313
20	52.840	323
30	46.621	333

Appendix -B

BULK DENSITY (g/cm³)		10 TON
Particle size	B	symbol
75	0.7355	111
90	0.684	112
150	0.6228	113
75	0.7825	121
90	0.7172	122
150	0.6516	123
75	0.8612	131
90	0.74815	132
150	0.7103	133

BULK DENSITY (g/cm³)		10 TON
Binder %	B	symbol
10	0.7355	111
20	0.7825	121
30	0.8612	131
10	0.684	112
20	0.7172	122
30	0.74815	132
10	0.6228	113
20	0.6516	123
30	0.7103	133

BULK DENSITY (g/cm³)		20 TON
Particle size	B	symbol
75	0.7376	211
90	0.6991	212
150	0.6285	213
75	0.7952	221
90	0.7405	222
150	0.6655	223
75	0.8894	231
90	0.74819	232
150	0.719	233

BULK DENSITY (g/cm³)		20 TON
Binder %	B	symbol
10	0.7376	211
20	0.7952	221
30	0.8894	231
10	0.6991	212
20	0.7405	222
30	0.74819	232
10	0.6285	213
20	0.6655	223
30	0.719	233

BULK DENSITY (g/cm³)		30 TON
Particle size	B	symbol
75	0.7536	311
90	0.7331	312
150	0.6351	313
75	0.8322	321
90	0.7791	322
150	0.6834	323
75	0.8981	331
90	0.7914	332
150	0.741	333

BULK DENSITY (g/cm³)		30 TON
Binder %	B	symbol
10	0.7536	311
20	0.8322	321
30	0.8981	331
10	0.7331	312
20	0.7791	322
30	0.7914	332
10	0.6351	313
20	0.6834	323
30	0.741	333

Appendix -B

LIPTON DYE TEST		10 TON
Particle size	(Abs.) at $\lambda=340$	symbol
75	0.16	111
90	0.282	112
150	0.439	113
75	0.2	121
90	0.439	122
150	0.65	123
75	1.168	131
90	1.326	132
150	2.233	133

LIPTON DYE TEST		10 TON
Binder %	(Abs.) at $\lambda=340$	symbol
10	0.16	111
20	0.2	121
30	1.168	131
10	0.282	112
20	0.439	122
30	1.326	132
10	0.439	113
20	0.65	123
30	2.233	133

LIPTON DYE TEST		20 TON
Particle size	(Abs.) at $\lambda=340$	symbol
75	0.015	211
90	0.083	212
150	0.15	213
75	0.073	221
90	0.097	222
150	0.172	223
75	0.563	231
90	0.924	232
150	1.933	233

LIPTON DYE TEST		20 TON
Binder %	(Abs.) at $\lambda=340$	symbol
10	0.015	211
20	0.073	221
30	0.563	231
10	0.083	212
20	0.097	222
30	0.924	232
10	0.15	213
20	0.172	223
30	1.933	233

LIPTON DYE TEST		30 TON
Particle size	(Abs.) at $\lambda=340$	symbol
75	0.013	311
90	0.042	312
150	0.092	313
75	0.068	321
90	0.07	322
150	0.142	323
75	0.099	331
90	0.120	332
150	1.553	333

LIPTON DYE TEST		30 TON
Binder %	(Abs.) at $\lambda=340$	symbol
10	0.013	311
20	0.068	321
30	0.099	331
10	0.042	312
20	0.07	322
30	0.120	332
10	0.092	313
20	0.142	323
30	1.553	333

Appendix -B

LIPTON DYE TEST		10 TON
Particle size	C_f	symbol
75	1.015	111
90	1.789	112
150	2.785	113
75	1.269	121
90	2.785	122
150	4.124	123
75	7.411	131
90	8.413	132
150	14.168	133

LIPTON DYE TEST		10 TON
Binder %	C_f	symbol
10	1.015	111
20	1.269	121
30	7.411	131
10	1.789	112
20	2.785	122
30	8.413	132
10	2.785	113
20	4.124	123
30	14.168	133

LIPTON DYE TEST		20 TON
Particle size	C_f	symbol
75	0.095	211
90	0.5266	212
150	0.9517	213
75	0.4631	221
90	0.6154	222
150	1.0913	223
75	3.572	231
90	5.862	232
150	12.265	233

LIPTON DYE TEST		20 TON
Binder %	C_f	symbol
10	0.095	211
20	0.4631	221
30	3.572	231
10	0.5266	212
20	0.6154	222
30	5.862	232
10	0.9517	213
20	1.0913	223
30	12.265	233

LIPTON DYE TEST		30 TON
Particle size	C_f	symbol
75	0.0824	311
90	0.266	312
150	0.583	313
75	0.431	321
90	0.444	322
150	0.901	323
75	0.6281	331
90	0.7614	332
150	9.854	333

LIPTON DYE TEST		30 TON
Binder %	C_f	symbol
10	0.0824	311
20	0.431	321
30	0.6281	331
10	0.266	312
20	0.444	322
30	0.7614	332
10	0.583	313
20	0.901	323
30	9.854	333

Appendix -B

CrO₃ OXIDE TEST		10 TON
Particle size	C_f (ppm)	symbol
75	1.448	111
90	1.853	112
150	45.191	113
75	1.68	121
90	4.808	122
150	67.323	123
75	2.491	131
90	8.612	132
150	85.399	133

CrO₃ OXIDE TEST		10 TON
Binder %	C_f (ppm)	symbol
10	1.448	111
20	1.68	121
30	2.491	131
10	1.853	112
20	4.808	122
30	8.612	132
10	45.191	113
20	67.323	123
30	85.399	133

CrO₃ OXIDE TEST		20 TON
Particle size	C_f (ppm)	symbol
75	0.463	211
90	0.579	212
150	4.171	213
75	0.521	221
90	1.332	222
150	7.589	223
75	1.7960	231
90	3.1286	232
150	24.044	233

CrO₃ OXIDE TEST		20 TON
Binder %	C_f (ppm)	symbol
10	0.463	211
20	0.521	221
30	1.7960	231
10	0.579	212
20	1.332	222
30	3.1286	232
10	4.171	213
20	7.589	223
30	24.044	233

CrO₃ OXIDE TEST		30 TON
Particle size	C_f (ppm)	symbol
75	0.1738	311
90	0.3476	312
150	1.9119	313
75	0.4519	321
90	1.1587	322
150	6.4889	323
75	1.448	331
90	1.969	332
150	9.31	333

CrO₃ OXIDE TEST		30 TON
Binder %	C_f (ppm)	symbol
10	0.1738	311
20	0.4519	321
30	1.448	331
10	0.3476	312
20	1.1587	322
30	1.969	332
10	1.9119	313
20	6.4889	323
30	9.31	333

Appendix -B

CrO₃ OXIDE TEST		10 TON
Particle size	(Abs.) at λ=460	symbol
75	0.025	111
90	0.032	112
150	0.78	113
75	0.029	121
90	0.083	122
150	1.162	123
75	0.043	131
90	0.148	132
150	1.474	133

CrO₃ OXIDE TEST		10 TON
Binder %	(Abs.) at λ=460	symbol
10	0.025	111
20	0.029	121
30	0.043	131
10	0.032	112
20	0.083	122
30	0.148	132
10	0.78	113
20	1.162	123
30	1.474	133

CrO₃ OXIDE TEST		20 TON
Particle size	(Abs.) at λ=460	symbol
75	0.008	211
90	0.01	212
150	0.072	213
75	0.009	221
90	0.023	222
150	0.131	223
75	0.031	231
90	0.054	232
150	0.415	233

CrO₃ OXIDE TEST		20 TON
Binder %	(Abs.) at λ=460	symbol
10	0.008	211
20	0.009	221
30	0.031	231
10	0.01	212
20	0.023	222
30	0.054	232
10	0.072	213
20	0.131	223
30	0.415	233

CrO₃ OXIDE TEST		30 TON
Particle size	(Abs.) at λ=460	symbol
75	0.003	311
90	0.006	312
150	0.033	313
75	0.0078	321
90	0.02	322
150	0.112	323
75	0.025	331
90	0.034	332
150	0.255	333

CrO₃ OXIDE TEST		30 TON
Binder %	(Abs.) at λ=460	symbol
10	0.003	311
20	0.0078	321
30	0.025	331
10	0.006	312
20	0.02	322
30	0.034	332
10	0.033	313
20	0.112	323
30	0.255	333

Appendix -B

CrO₃ OXIDE TEST		10 TON
Particle size	%ε	symbol
75	98.552	111
90	98.147	112
150	54.809	113
75	98.32	121
90	95.192	122
150	32.677	123
75	97.509	131
90	91.388	132
150	14.601	133

CrO₃ OXIDE TEST		10 TON
Binder %	%ε	symbol
10	98.552	111
20	98.32	121
30	97.509	131
10	98.147	112
20	95.192	122
30	91.388	132
10	54.809	113
20	32.677	123
30	14.601	133

CrO₃ OXIDE TEST		20 TON
Particle size	%ε	symbol
75	99.537	211
90	99.421	212
150	95.829	213
75	99.479	221
90	98.668	222
150	92.44	223
75	98.204	231
90	96.8714	232
150	75.956	233

CrO₃ OXIDE TEST		20 TON
Binder %	%ε	symbol
10	99.537	211
20	99.479	221
30	98.204	231
10	99.421	212
20	98.668	222
30	96.8714	232
10	95.829	213
20	92.44	223
30	75.956	233

CrO₃ OXIDE TEST		30 TON
Particle size	%ε	symbol
75	99.826	311
90	99.6524	312
150	98.0881	313
75	99.5481	321
90	98.8413	322
150	93.5111	323
75	98.552	331
90	98.013	332
150	85.226	333

CrO₃ OXIDE TEST		30 TON
Binder %	%ε	symbol
10	99.826	311
20	99.5481	321
30	98.552	331
10	99.6524	312
20	98.8413	322
30	98.013	332
10	98.0881	313
20	93.5111	323
30	85.226	333

Appendix -B

LIPTON DYE TEST		10 TON
Particle size	%ε	symbol
75	98.985	111
90	98.211	112
150	97.215	113
75	98.731	121
90	97.215	122
150	95.876	123
75	92.589	131
90	91.587	132
150	85.832	133

LIPTON DYE TEST		10 TON
Binder %	%ε	symbol
10	98.985	111
20	98.731	121
30	92.589	131
10	98.211	112
20	97.215	122
30	91.587	132
10	97.215	113
20	95.876	123
30	85.832	133

LIPTON DYE TEST		20 TON
Particle size	%ε	symbol
75	99.905	211
90	99.473	212
150	98.834	213
75	99.5369	221
90	99.384	222
150	98.9	223
75	96.428	231
90	94.138	232
150	87.735	233

LIPTON DYE TEST		20 TON
Binder %	%ε	symbol
10	99.905	211
20	99.5369	221
30	96.428	231
10	99.473	212
20	99.384	222
30	94.138	232
10	98.834	213
20	98.9	223
30	87.735	233

LIPTON DYE TEST		30 TON
Particle size	%ε	symbol
75	99.917	311
90	99.734	312
150	99.048	313
75	99.569	321
90	99.556	322
150	99.099	323
75	99.3719	331
90	99.238	332
150	90.146	333

LIPTON DYE TEST		30 TON
Binder %	%ε	symbol
10	99.917	311
20	99.569	321
30	99.3719	331
10	99.734	312
20	99.556	322
30	99.238	332
10	99.048	313
20	99.099	323
30	90.146	333

Appendix -B

HARDNESS		10 TON
Particle size	HB(Kg/mm²)	symbol
75	50	111
90	45	112
150	41	113
75	60	121
90	57	122
150	53	123
75	66	131
90	65	132
150	63	133

HARDNESS		10 TON
Binder %	HB(Kg/mm²)	symbol
10	50	111
20	60	121
30	66	131
10	45	112
20	57	122
30	65	132
10	41	113
20	53	123
30	63	133

HARDNESS		20 TON
Particle size	HB(Kg/mm²)	symbol
75	56	211
90	52	212
150	47	213
75	65	221
90	60	222
150	57	223
75	73	231
90	69	232
150	67	233

HARDNESS		20 TON
Binder %	HB(Kg/mm²)	symbol
10	56	211
20	65	221
30	73	231
10	52	212
20	60	222
30	69	232
10	47	213
20	57	223
30	67	233

HARDNESS		30 TON
Particle size	HB(Kg/mm²)	symbol
75	59	311
90	56	312
150	49	313
75	68	321
90	66	322
150	61	323
75	77	331
90	76	332
150	74	333

HARDNESS		30 TON
Binder %	HB(Kg/mm²)	symbol
10	59	311
20	68	321
30	77	331
10	56	312
20	66	322
30	76	332
10	49	313
20	61	323
30	74	333

C _o (CrO ₃)	Abs.	blank
100(ppm)	1.628	000
50 (ppm)	0.863	000