

REGENERATION OF POWDERED ACTIVATED CARBON EXHAUSTED DURING GLYCERINE BLEACHING

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إعادة تنشيط الكربون الناعم المستخدم في عملية تبييض الجلسرين

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خلاصة

يتناول هذا البحث دراسة عملية لإعادة تنشيط الكربون الناعم المستخدم والمستهك نشاطه في عمليات إزالة اللون والرائحة للجلسرين المستخدم في الأفراس الصناعية والطبية .
وقد شملت الدراسة عملية إستخلاص الجلسرين العالق بالكربون المستهلك قبل إعادة تنشيطه وحددت الظروف المثلى لعملية الإستخلاص والتي عن طريقها أمكن إستخلاص كمية من الجلسرين تعادل 36% من وزن الكربون المستهلك .
وهذه الظروف تتمثل في خلط الكربون المستهلك مع الماء بنسبة 1 : 4 وزناً والتقليب الجيد مع التسخين حتى 80 - 85 درجة مئوية لمدة نصف ساعة . كما حددت هذه الدراسة أنسب الظروف لعملية إسترجاع نشاط الكربون والتي من خلالها أمكن إعادة تنشيط الكربون المستهلك بحيث أصبح نشاطه يمثل 80% من نشاط الكربون الأصيل . وتمثل هذه الظروف في تسخين الكربون في بيئة مغطاه حتى 500 درجة مئوية لمدة ساعة .

ABSTRACT: Powdered activated carbon exhausted during glycerine bleaching can be regenerated and reused again. Glycerine retained by the exhausted carbon can be recovered before the regeneration process. Optimum glycerine recovery conditions were found to mix, efficiently, 4 parts (by weight) of wash water per part of exhausted carbon for half an hour at 80-85°C. About 36% by weight from exhausted carbon was recovered as glycerol by this technique.

Above 85% of the activity of the exhausted powdered activated carbon can be restored by thermal regeneration. Best results were obtained on heating the exhausted powdered activated carbon for an hour at a temperature of 500°C or higher.

INTRODUCTION

The aim of glycerine industry is to recover glycerine from the lyes of fat splitting or soap production and to forward it to the market in one of the following forms: crude glycerine, 80% glycerol brown coloured, dynamite glycerine, 95-98% glycerol pale-straw coloured, industrial white glycerine made by bleaching or decolourising dynamite glycerine and chemically pure glycerine or pharmaceutical glycerine which is about 99% glycerol and water white.

The various processes involved in recovering concentrated glycerine from the lyes include treatment, evaporation, distillation and bleaching in the case of high quality requirements. In bleaching or decolourising traces of pigments and odours left in the distilled glycerine are removed by adsorption on powdered activated carbon. This is accomplished by mixing distilled glycerine batchwise with fine-grained activated carbon and agitating them in a closed mixing vessel at a temperature of approximately 80°C so that both components form a homogeneous mixture. The required quantity of powdered activated carbon is about 0.1-0.5% by weight of glycerine required. The exhausted powdered activated carbon is then separated by filtration in a filter press [1,2].

The exhausted powdered activated carbon is discarded with the glycerine retained in it, as there is no benefit from it. Some soap companies pay for its discarding, while others suffer from its huge amounts which need a large area for dumping without any present or future use.

In Egypt, glycerine industry needs about 20 tons per year of powdered activated carbon. This quantity is completely imported from abroad at a total cost of about 10 000 Egyptian pounds per ton, (prices of 1988) [6]. The aim of the undertaken work is to investigate the regeneration of the exhausted activated carbon used in glycerine bleaching, in order to reuse it again and hence to save part of that imported. At the same time the glycerol retained within the powdered activated carbon can be restored.

Regeneration can be achieved by thermal, chemical, hot gas, solvent, or biological methods. Generally, methods other than thermal will not be effective enough if a mixture of organic chemicals has been adsorbed [3,4,11].

Only a portion of the sorbed materials will be removed by a given solvent, hot gas, chemical or biological regeneration procedure. Therefore, performance of the carbon will continually decrease on successive regenerations and after few regenerations, the carbon will have to be discarded [5].

Thermal regeneration, the specific subject of this work, is the most versatile of the techniques mentioned above, and because most powdered and granular carbon treatment systems remove a complex mixture of organics, it is the most widely used,

Experimental:

Experimental work was divided into two main steps. The first one was to investigate the optimum operating conditions for glycerol recovery from the exhausted powdered activated carbon. The second step was to determine the best conditions for the regeneration of the washed exhausted activated carbon.

Glycerol recovery was performed batchwise by adding a certain volume of water to 50 gm of the exhausted carbon cake and agitation for a certain time in a closed mixing vessel immersed in a water bath. The speed of the anchor impeller was about 500 rpm. Glycerol solution was separated by filtration under reduced pressure using 0.7 μ m filter paper.

Filter cake obtained from washing and filtering the exhausted powdered activated carbon was transferred to an iron crucible covered with lid and regenerated in muffle furnace then transferred into a desiccator. The regenerated carbon samples were taken from the desiccator and stored in tight sealed containers for subsequent use.

Adsorption capability of the regenerated powdered activated carbon was assessed by comparing the adsorption characteristics of the regenerated and the genuine powdered activated carbon with respect to the decolourization of methylene blue solution, decolourizing of distilled glycerine, and adsorption of acetic acid. These were performed according to Japanese Industrial Standard JIS-K-1470 [7].

Decolourization of methylene blue solution was measured colourimetrically by measuring the absorbancy on a spectrophotometer (Shimadzu model UV-120-02, Japan) at a wave length of 665 nm. Adsorption capability for acetic acid was performed through back titration of the residual acetic acid against 0.1 M NaOH solution.

RESULTS AND DISCUSSION:

Results obtained from glycerol recovery are given in Table (1) and are illustrated in Figures 1 and 2 these results show the effect of the volume of wash water per 50 gm of the exhausted activated carbon, operating temperature and contact time upon the glycerol content in wash water. These results reveal the following:

- a) Glycerol recovery increases with the increase of the volume of wash water for a specific contact time and operating temperature.
- b) Glycerol recovery increases with the increase in the operating temperature, while the other factors are held constant.
- c) Increasing contact time increases the quantity of glycerol recovered. The threshold value of contact time is about 0.5 hour.

Best results for glycerol recovery were obtained when mixing one part from the exhausted carbon with four parts of water at 80-85°C for half an hour. This facilitates the recovery of more than 36% (by weight) of the original exhausted carbon as glycerol.

Eight filter cakes obtained from washing and filtering exhausted powdered activated carbon were thermally regenerated as given in Table 2.

The particule size for these samples and for the genuine sample were found to be below $53 \mu\text{m}$.

Results of decolourizing of methylene blue solution and bleaching of glycerol by the regenerated carbon samples are also noted in Table 2.

The following can be deduced from Table 2:

- 1) Sample number 6 gives the best decolourization action followed by samples 7,8 and 4 respectively.
- 2) More than 85% of the exhausted adsorption activity of powdered activated carbon can be restored by thermal regeneration under the operating conditions specified for sample 6 (heating for 1.5 hrs at 700°C).
- 3) Best results for thermal regeneration can be obtained by heating the washed carbon sample for one hour at a temperature of 500°C or higher.

Adsorption of acetic acid on samples 6 and 4 was assessed by determining the adsorption isotherms for them and for the genuine powdered activated carbon. The resulting adsorption isotherms are shown in Fig.3, which are compatible with those for adsorption on solid surfaces from solutions [8,9,12].

This figure reveals the following:

- 1) Regenerated and genuine powdered activated carbon have the same adsorption characteristics.
- 2) At low concentrations of the adsorbate, the regenerated carbon and the genuine carbon has almost the same adsorption capacity.
- 3) Differences in reactivity between the various samples appear at moderate and high concentrations.
- 4) The maximum quantity of adsorbate per unit mass of adsorbent is different for each sample. For sample 6, this limiting value - which indicates that the adsorbing surface has been completely saturated - is only 82% of that for the genuine carbon while for sample 4 it is only 73.3% of that for the genuine carbon.

The resulting adsorption isotherms were analyzed with the various isotherm equations [8,9,12]. The Freundlich equation successfully describes the ascending branch of these isotherms only (Fig.3-b) which is the general case for adsorption from solutions [12].

CONCLUSION

- 1) Powdered activated carbon exhausted in bleaching of glycerine can be regenerated thermally. The regenerated and the genuine active carbon have the same adsorption

- 2) Glycerol retained by this exhausted carbon can be restored before the regeneration process. Glycerol restored represents about 36% (by weight) of the original sample.
- 3) The best operating conditions for glycerol recovery were to add 4 parts (by weight) of wash water per each part of the exhausted carbon cake then efficient mixing by a suitable agitator for half an hour at a temperature of 80-85°C.
- 4) Best results for thermal regeneration were obtained on heating the exhausted carbon for one hour at a temperature of 500°C or higher. Above 85% of the activity of the exhausted powdered activated carbon was restored by this technique.

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Table (1): Results of the glycerol recovery experiments

Volume of wash water, ml	Temp, (°C)	Time, (hr)			
		0.25	0.5	1	2
		GLYCEROL RECOVERED, (gm)			
100	33	3.82	6.71	7.02	7.38
	60	4.71	7.63	7.90	8.08
	80	5.78	10.72	10.99	11.29
150	33	5.37	9.71	10.37	10.59
	60	6.78	10.07	10.11	10.92
	80	8.43	14.23	14.43	14.61
200	33	6.9	12.76	13.32	13.82
	60	8.92	13.20	13.56	14.06
	80	9.68	17.88	18.12	18.46
300	33	9.06	15.87	17.66	18.33
	60	11.01	16.77	19.95	20.13
	80	13.83	20.97	21.15	21.36

Table (2): Operating conditions for washing and thermal regeneration of the exhausted powdered activated carbon samples versus their decolourizing power ratio.

Sample No.	Washing process			Thermal regeneration process.		Decolourizing power ratio, R*	
	V, ml.	Temp °C	Time, hr	Time, hr	Temp., °C	R ₁	R ₂
1	200	80	0.5	0.5	350	0.060	0.065
2	200	80	0.5	1.5	350	0.068	0.072
3	200	80	0.5	0.5	500	0.084	0.080
4	200	80	0.5	1.0	500	0.801	0.823
5	200	80	0.5	0.5	700	0.290	0.323
6	200	80	0.5	1.5	700	0.866	0.871
7	100	33	0.5	1.0	700	0.853	0.833
8	200	33	0.5	1.0	700	0.846	0.865

* R = (Decolourizing power of the sample ÷ Decolourizing power of genuine powdered activated carbon).

Subscript (1) refers to methylene blue decolourization while subscript (2) refers to distilled glycerol decolourization.

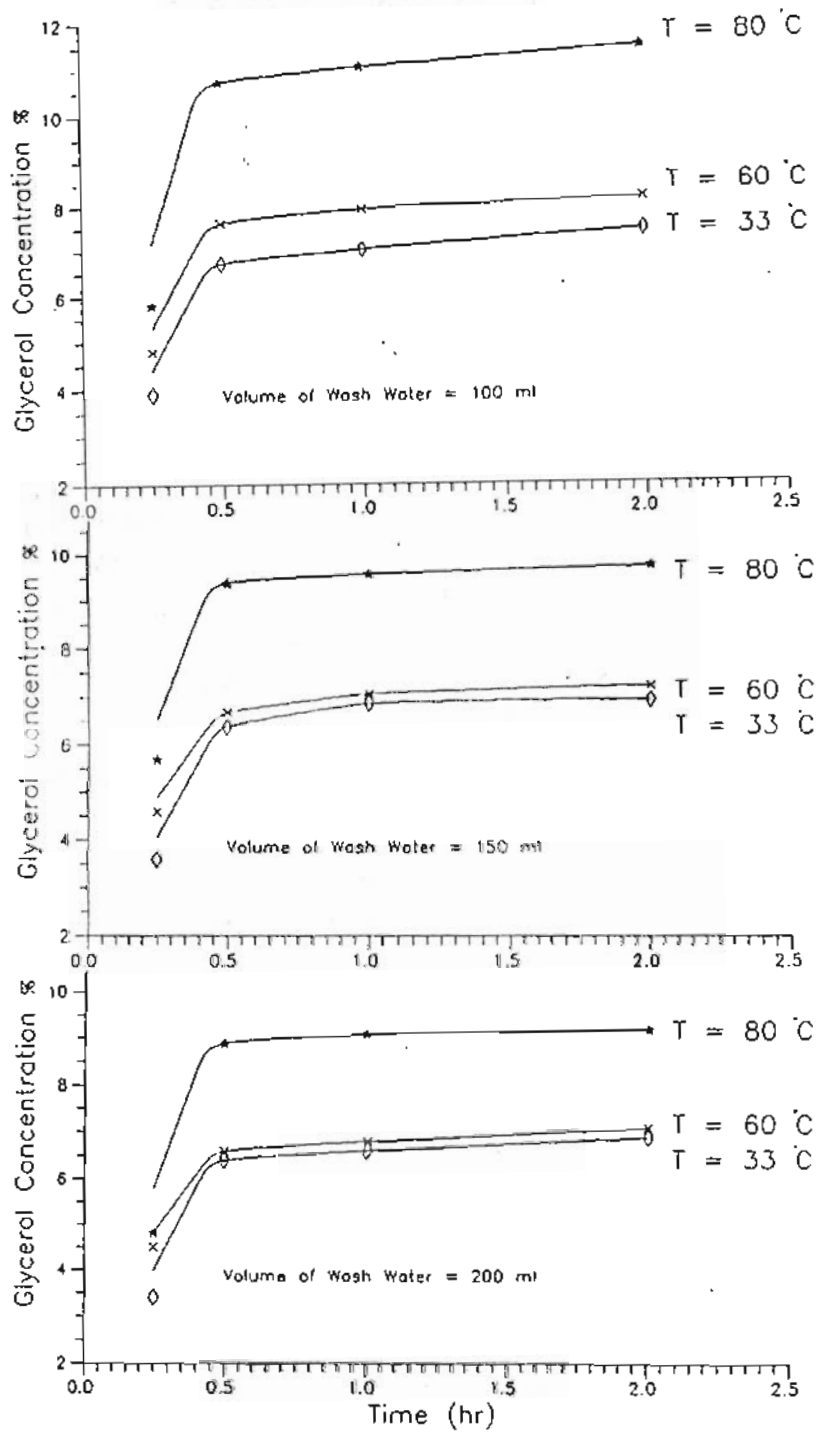


Fig. (1): Glycerol recovery as a function of operating temperature and volume of wash water.

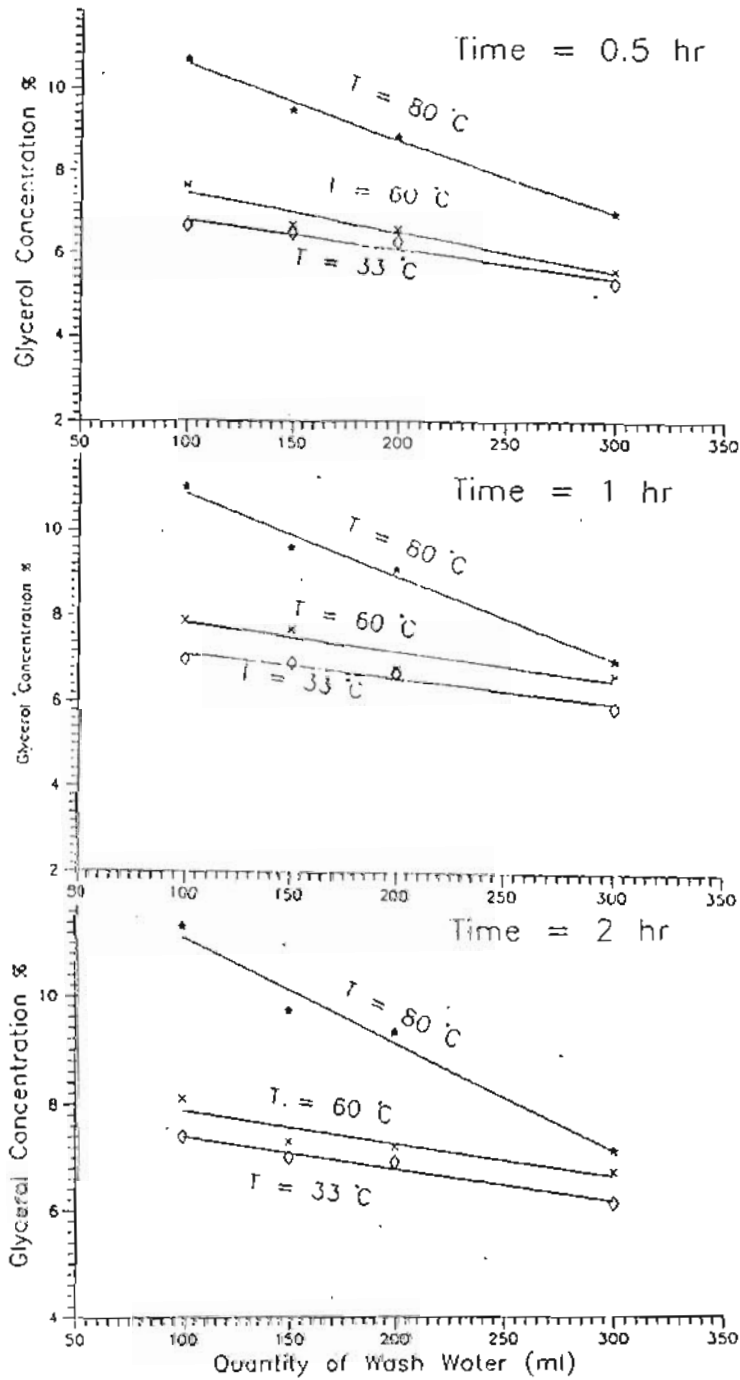


Fig (12): Glycerol recovery as a function of contact time and quantity of wash water.

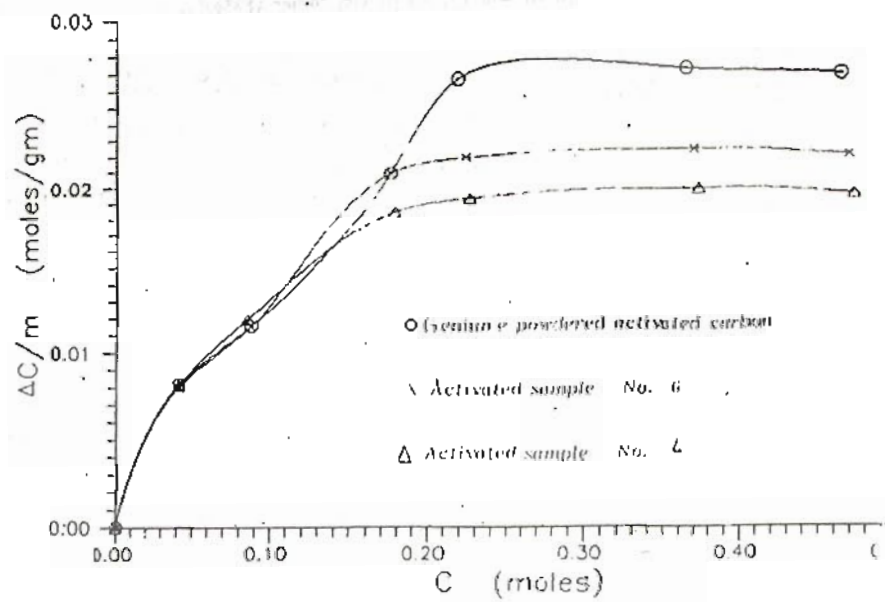


Fig. 13.a): Acetic acid adsorption data I (at 25 °C).

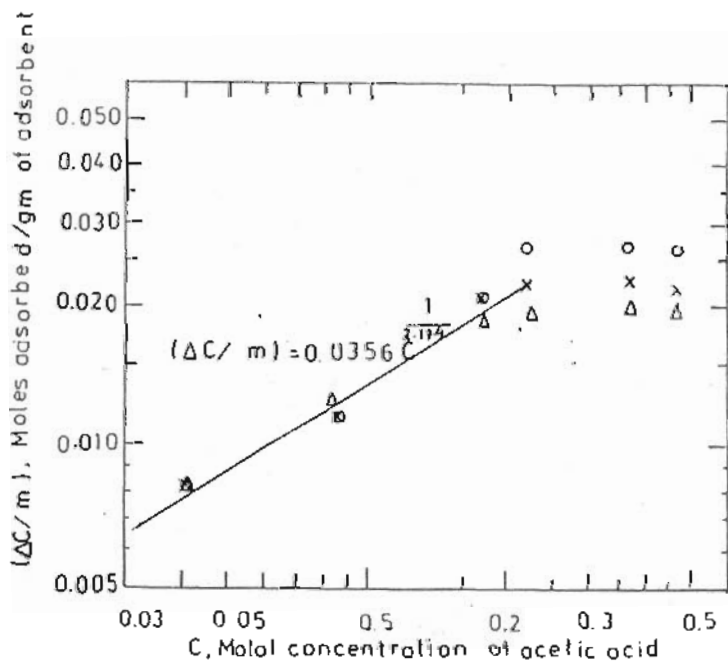


Fig.13.b): Logarithmic plot of acetic acid adsorption (at 25 °C)