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Investigation of the Potential of Activated and Magnetic Activated Carbon Produced from Turkish Lignite as Gold Adsorbents

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ABSTRACT

Activated carbon was prepared from Tuncbilek-Kutahya (Turkey) lignite (LAC) which was chosen as a precursor due to its availability and low-cost. The precursor was impregnated with distilled water and KOH and was selected in 1:4 ratios. A Magnetic Activated Carbon (MAC) was prepared by 0.5 g of the modified LAC in 20 mL aqueous solution containing 4 g $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. The ability of the activated carbons to adsorb gold ions from aqueous solution by adsorption was investigated. The BET surface area values of the activated carbons ranged from 667-928 $\text{m}^2 \text{g}^{-1}$. The effect of particle size on the activated carbon had very small effect on the specific surface area because of internal pore structure development. The effects of various experimental parameters such as initial gold concentration, pH and adsorbent dosage were investigated in a batch adsorption technique. The percentage of gold adsorption onto Commercial Activated Carbon (CAC), LAC and MAC showed the same trend, they adsorbed more than 80% of gold in the first 15 min and nearly 100% in next 45 min. On the other hand, the coarser carbon, CAC-1, adsorbed nearly 30% of gold in first 15 min and 65% in next 45 min due to the particle size effect.

Key words: Gold, magnetic activated carbon, BET surface area, adsorption

INTRODUCTION

Activated Carbon (AC) is the commercial name for a carbonaceous material that is a highly porous and predominantly amorphous solid, high surface area and the surface functional groups consists of hydrophobic grapheme and hydrophilic surface layer. The unique characteristics of commercial activated carbon can be used as adsorbents, catalyst and catalyst support (McDougall and Hancock, 1980; Bansal *et al.*, 1988). To meet the demands, low-cost activated carbons are produced from a wide variety of raw materials (sugar cane bagasse, wood and agricultural residues such as coconut shell, fruit stones) by two methods physical and chemical activation or a combination of both (Evans *et al.*, 1999; Hayashi *et al.*, 1995; Soleimani and Kaghazchi, 2008).

Due to unique characteristics of activated carbon, it can be used as adsorbent to treat water and industrial wastewater for removing organic and inorganic pollutants. Activated carbon application has been increasing in the mining industry for extracting gold and silver from cyanide solution (Soleimani and Kaghazchi, 2008). The first and foremost investigation process for leaching by chlorine had been investigated by Bansal and Goyal (2005) for recovery of gold by adsorption on wood charcoal and this process patented. However, first serious attempt was made by Zadra *et al.*

(1952) used granular activated carbon to cyanide pulp Carbon-In-pulp (CIP) and recovered the gold loaded carbon from solution by screening. Their studies become milestone in the history of gold industry. Afterwards, in response to the need for the recovery of gold from leaching solutions, generally low-cost adsorbents have been used to adsorb the leaching solution and followed by elution of adsorbed gold from loaded activated carbon (Ladeira *et al.*, 1993; Ibrado and Fuerstenau, 1994).

Some researchers in their finding claimed that fine particle gain magnetic properties, the obtained Magnetic Activated Carbon (MAC) can be distant easily by a wet magnetic separator, as conflicting to the traditional screening technology. This decreases carbon losses, capital cost and operating cost (Munoz *et al.*, 2002; Miller *et al.*, 2001; Miller *et al.*, 2004; Munoz, 2006). As known that extensive study has been carried out to understand the mechanism of gold adsorption by activated carbon. The mechanism is best described by the ion-pairs $\{Mn+[Au(CN)_2]^- \}_n$ are adsorbed onto the carbon surface devoid of chemical change (Marsden and House, 1993). The main objective of the present study was to produce low-cost activated carbon namely activated and magnetic activated carbon from Turkish Lignite (Tuncbilek-Kutahya) and also study of the applicability and suitability of the obtained low-cost activated and magnetic activated carbons in gold adsorption and elution process.

MATERIALS AND METHODS

In this present study, $KAu(CN)_2$ (Johnson Matthey), KCN, NaCN, H_3BO_3 , $CaCl_2$, KCl and NaOH chemical reagents were used. All the reagents were A.R. grade and used without further treatment. Activated carbon used in this study was supplied from Calgon DG-13/Calgon Corporation Company and the prepared activated carbon from Turkish lignite.

Preparation of Activated Carbon (LAC): Activated carbon was prepared from Tuncbilek-Kutahya (Turkey) lignite which was chosen as a precursor due to its availability and low-cost. The lignite sample was crushed and sieved to -60+40 mesh size fraction. The precursor was impregnated with distilled water and KOH. The impregnation ratio (i.e., lignite/KOH) was selected as 1:4. The blend was dried at $105^\circ C$ and then it was heated in tube furnace at $800^\circ C$ for 1 h under N_2 (100 mL min^{-1}) atmosphere with a $10^\circ C\text{ min}^{-1}$ heating rate. Afterwards, the sample was cooled under flowing N_2 and washed with diluted HCl. In order to remove chloride ions from activated carbon, the sample rewashed with distilled water several times until filtrate gives no reaction with $AgNO_3$ and dried at $105^\circ C$. The activated carbon was ground in a standard ring mill and sieved under 200 mesh sizes ($75\text{ }\mu m$). The obtained material was denoted as LAC.

Preparation of Magnetic Activated Carbon (MAC): Tuncbilek lignite (LAC) was obtained locally. LAC was cleaned and washed thoroughly to remove water-soluble substances. The material was acidified using concentrated nitric acid and subsequently allowed to soak for 24 h at room temperature. Thereafter, the sample material was boiled at $80^\circ C$ for 3 h followed by cooling. The 0.5 g of modified LAC was soaked in 20 mL aqueous solution containing 4 g $Fe(NO_3)_3 \cdot 9H_2O$ and the mixture was placed into ultrasonic bath to disperse LAC. The mixture was filtrated and washed thoroughly with distilled water, till the uniform characteristics of both filtering and filtered water (with respect to color, pH and turbidity) was obtained and was dried and subsequently heated in tube furnace at $750^\circ C$ for 3 h in the presence of nitrogen. The obtained material was Magnetic Activated Carbon (MAC) (Yang *et al.*, 2008).

Commercial Activated Carbon (CAC): In order to evaluate the applicability of LAC and MAC in extraction of gold, commercial activated carbon was used as a standard adsorbent to compare the experiment results obtained from LAC and MAC. Commercial activated carbon was supplied by Calgon Corporation (Calgon GRC 22), two different sizes namely original size, -7+12 mesh (CAC-1) and milled activated carbon, -200 mesh (CAC-2).

Instrumentation: Gold analysis was done by a Plasma-400 ICP-AES [Inductively-Coupled Plasma (ICP)-Atomic Emission Spectrometer (AES)] instrument and atomic absorption spectrophotometer with an air-acetylene flame and an absorbance peak at a wavelength of 242.8 nm. The BET surface area, total pore volume, average pore radius, micropore area were obtained by surface analyzer of model Tri Star 3000 (Micromeritics, USA) was used to measure nitrogen adsorption isotherm at 77 K in the range of relative pressures from 10^{-6} to 1. Before measurement, the sample was degassed at 400°C for 2 h. Mesopore volume was determined by subtracting the micropore volume from total pore volume.

Adsorption capacity: To carry out isotherm studies, synthetic gold solutions (100 ppm) were prepared from $\text{KAu}(\text{CN})_2$ at pH 10 buffered by a borate solution (3.09 g of boric acid and 3.73 g of potassium chloride, 2 g of NaOH and DI-water and then 0.1462 g of $\text{KAu}(\text{CN})_2$). The solution was equilibrated in shaker at room temperature for 24 h at 200 rpm and followed by filtration of the adsorbent and subsequent analysis of filtrate for gold concentration. The percentage removal of gold and equilibrium adsorption uptake, q_e (mg g^{-1}) was calculated using the following relationships:

$$\text{Removal (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$\text{Amount adsorbed (mg g}^{-1}\text{)} q_e = \frac{(C_0 - C_e)V}{w} \quad (2)$$

where, C_0 is the initial adsorbate concentration (mg L^{-1}), C_e the equilibrium adsorbate concentration (mg L^{-1}), V is the volume of the solution (L) and w is the mass of the adsorbent (g).

Gold adsorption capacity was found by using Freundlich isotherm to understand the possibility of multilayer adsorption and non-linear energy distribution for the adsorption sites of the same adsorbents. Freundlich equation is expressed as:

$$C_e = K C_s^{1/n} \quad (3)$$

where, 'K' and 'n' are the measures of adsorption capacity and intensity of adsorption. 'C_e' is the amount of phenol adsorbed per unit mass of adsorbent and 'C_s' is the equilibrium concentration in mg L^{-1} .

Adsorption rate: To optimize the dosage of adsorbent per unit mass of adsorbate, gold solution was added to different dosages of adsorbent still the equilibrium was attained. The mass of the adsorbent dosages ranging from 0.1-0.5 g added to 100 mL of 100 ppm gold solution containing reactor. The kinetics of analyzing adsorptive uptake determined adsorption of gold from aqueous solution at different intervals. The adsorption rate was calculated as follows:

$$t/(X/M) = (1/M)t + 1/R \quad (4)$$

where, X/M represents the carbon loading in mg Au/g of carbon, t is the time and M and R are reciprocal of the slope and intercept at zero time, respectively. High R -values indicate that gold adsorption onto activated carbon is very fast. In the present study the rate control mechanism have been studied by using intra-particle diffusion equation as follows (Weber and Morris, 1963):

$$q_t = k_{int} \times t^{1/2} + C \quad (5)$$

where, C is the intercept and k_{int} is the intra-particle diffusion rate constant ($\text{mg g}^{-1} \text{min}^{1/2}$). The intra particle diffusion rate obtained from the plots q_t vs. $t^{1/2}$.

Elution procedure: The elution process, sometimes referred to as “stripping” is a reversal of adsorption. In conventional stripping systems, desorption may takes place relatively at high temperature ($90\text{-}120^\circ\text{C}$) and required long elution period (12-72 h) (Zadra, 1950; Zadra *et al.*, 1952; Davidson and Duncanson, 1977; Davidson *et al.*, 1979). Gold desorption from loaded activated carbon was carried by hot elution and pressure desorption:



In the present batch desorption studies, two elution methods were employed to desorb gold from loaded activated carbon by Atmospheric Zadra Elution and Filter Press. Carbon loading was carried using 250 mg L^{-1} gold concentration as $KAu(CN)_2$ at pH 10 (buffered by borate solution). One gram of activated carbon was added to the 250 mg L^{-1} gold concentration solution and was soaked for 24 h at 200 rpm. Solution was filtered and gold concentration was determined by using absorption spectrophotometer. The quantity of gold recovered was calculated by following equation:

$$\text{Carbon loading (kg L}^{-1}\text{)} = \frac{([Au]_s^0 - [Au]_s^{24}) \times S_v}{M_c \times 1000} \quad (7)$$

where, $[Au]_s^0$ and $[Au]_s^{24}$ are initial and final gold concentration (ppm), respectively. S_v is the solution volume (mL) and M_c is the mass of adsorbate (g).

Atmospheric Zadra elution: The loaded activated carbon was thoroughly washed with de-ionized water dried and stored for further use. Zadra solution was prepared by taking NaCN 0.5%, 1 NaOH solution 1% and pH of the Zadra solution just between 12 and 13 (Marsden and House, 1993; Marsden and House, 2006; John and Fast, 1989). The batch desorption studies were conducted on loaded activated carbons (placed in the Zadra solution at $(98\text{-}100^\circ\text{C})$ and at atmospheric pressure over a 48 h exposure in the closed system) and the percent of gold eluted was calculated by following equation:

$$\text{Gold eluted (\%)} = \frac{A-B}{A} \times 100 \quad (8)$$

where, A and B are the amount of gold on activated carbons before and after elution, respectively.

Filter press elution: The second elution method (i.e., filter press method) was developed by, Davidson and Anglo American Research Laboratory [AARL]. The 3 g activated carbon was presoaked in 100 mL of Davidson solution (10% KOH and 5% K_2CO_3 , the pH of the solution is 13) at room temperature for 30 min (Davidson, 1974). Then, tap water (at 98°C) was added to the reactor to make one liter of solution. The solution was poured in camber of a filter press, the camber was closed and a pressure of (80 psi) was applied. The test was repeated 4 times. The stripped solution was collected and a sample was stored in sample vials for further gold analysis.

RESULTS AND DISCUSSION

Characterization of pore structure of activated carbons: Table 1 shows the characteristics of the activated carbons (namely the BET surface area (S_{BET}), external surface area (including mesopores and macropores area (S_{ext}), micropores surface area (S_{mic}), total pore volume (V_t) and average pore diameter (D_p). It was found that the activated carbons had a remarkable BET surface area which was primarily contributed by micropores. The average pore diameters for prepared activated carbon were between 2.16 and 2.44 nm, indicative micro-pores character. It appeared that activated carbons were dominantly micropores.

N_2 adsorption isotherms of LAC and MAC are shown in Fig. 1. It can be classified as type 1 which is characteristic of adsorbents having small pores as per IUPAC classification. N_2 adsorption isotherms, surface area and porosity values of LAC and MAC values less than that of CAC-1 and CAC-2. It can be attributed to adsorption of Fe_3O_4 by activated carbon. The results were supported by Yang *et al.* (2008) and Oliveira *et al.* (2002).

Table 1: Surface area and porosity values of the activated carbons

Code	S_{BET}	S_{mic}	S_{meso}	V_t	V_{mic}	V_{meso}	D_p (nm)
	-----($m^2 g^{-1}$)-----			-----($cm^3 g^{-1}$)-----			
CAC -1	906	794	112	0.490	0.42	0.070	2.16
CAC -2	928	807	121	0.504	0.44	0.064	2.17
LAC	947	825	122	0.580	0.43	0.150	2.44
MAC	667	581	86	0.270	0.21	0.060	2.28

D_p : 4V/A by BET

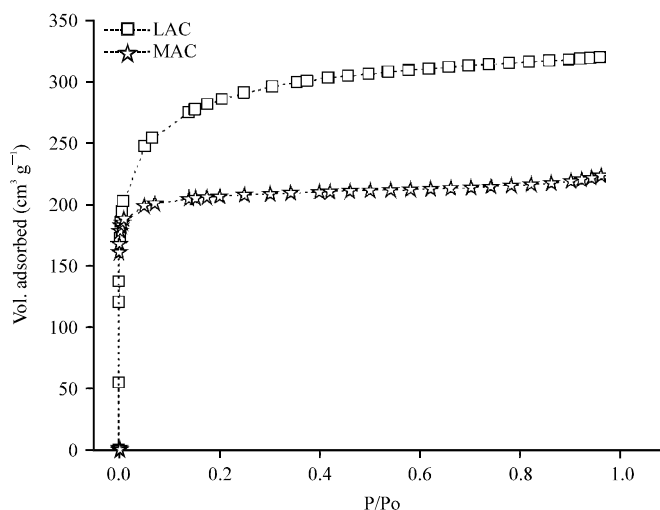


Fig. 1: Adsorption isotherm of nitrogen at 77 K for LAC and MAC

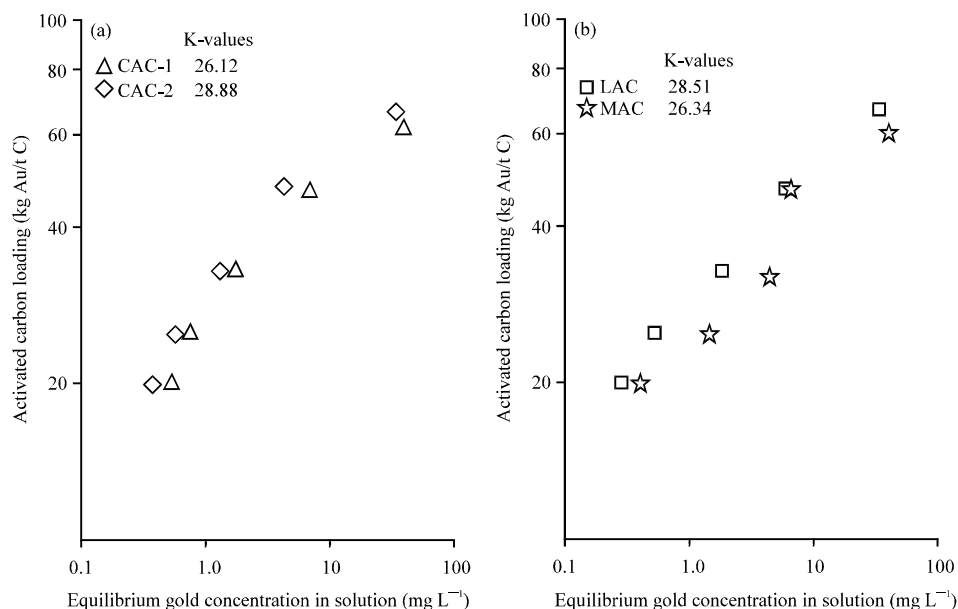


Fig. 2(a-b): Gold adsorption capacities of the activated carbons

Adsorption isotherm capacity: The adsorption isotherm experiment results for four adsorbents are shown in Fig. 2. The results indicate that the adsorption capacities (K-values) were almost same for CAC-2 and LAC which had same particle size. Although, CAC-1 and MAC had same K-values but less than that of CAC-2 and LAC, these values are greater than the expected minimum value (greater than 25 kg Au/t C) of activated carbons used in the industry (Shipman, 1994). It shows that modification process did not significantly affect the adsorption capacity of the adsorbents. It can be conclude from the results, the activated carbons (LAC and MAC) prepared from Tunçbilek lignite have good loading capacities. In addition to that, by comparing the K-value of CAC-1 and that of CAC-2, it could be clearly seen that K-values increased while decreasing the particle size of activated carbon (Elnathan, 2007; Munoz, 2006).

Adsorption rate: Figure 3 shows the results of the gold adsorption kinetics of the activated carbons. The adsorption capacity of CAC-2, LAC and MAC follows same trend. More than 80% of gold has been adsorbed within first 15 min and in next 45 min remaining 20% adsorption of gold onto the adsorbate. On the other hand, CAC-1 (the coarser carbon) only 30% of gold has been adsorbed in the first 15% and only 65% in the next 45 min. McArthur *et al.* (1987) and Shipman and Jativa (1987) in their studies found that, 60% gold were removed in the first 1 h. It can be concluded from the results, the adsorption capacity of the four adsorbents was due to intra-particle adsorption.

Figure 4 shows the gold adsorption rate and R²-values. The adsorption rate increased with decreasing particle size. Since, the size has a large effect on the mean pore length with in the carbon particles despite only a small increase in net surface area (Marsden and House, 1993). In addition, the adsorption rate of LAC and CAC-2 had higher adsorption rate (higher R²-values 8.09 and 7.69, respectively) and exhibiting great similarity with each other but MAC (R²-value 5.38) had lower adsorption rate than LAC and CAC-2 despite of the same particle size. On the other hand, the adsorption rate of MAC was nearly 11 times greater than that of CAC-1, because of particle size effect (Munoz, 2006; Marsden and House, 2006; Elnathan, 2007; Fleming and Nicol, 1984).

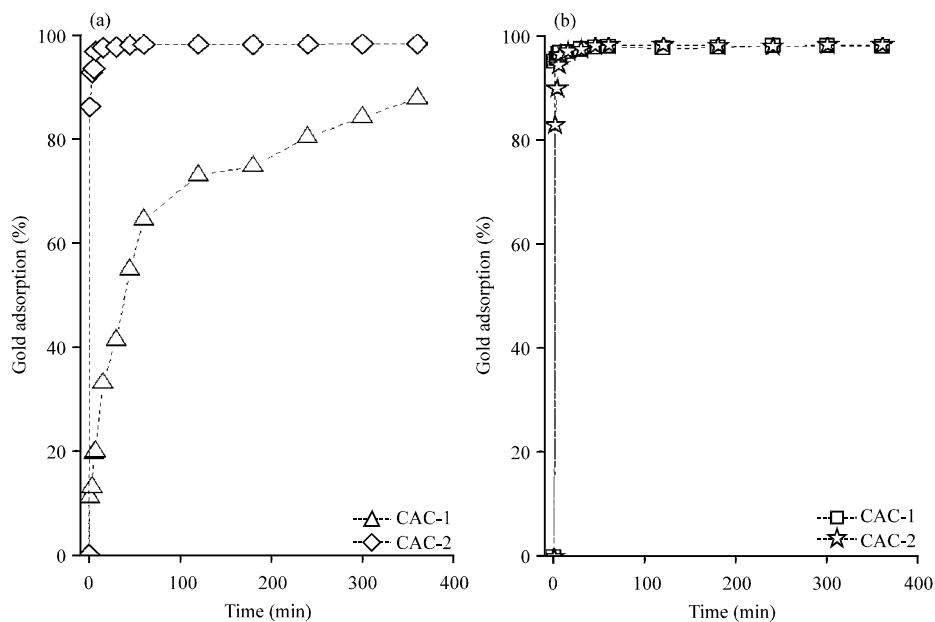


Fig. 3(a-b): Gold adsorption kinetics of activated carbons

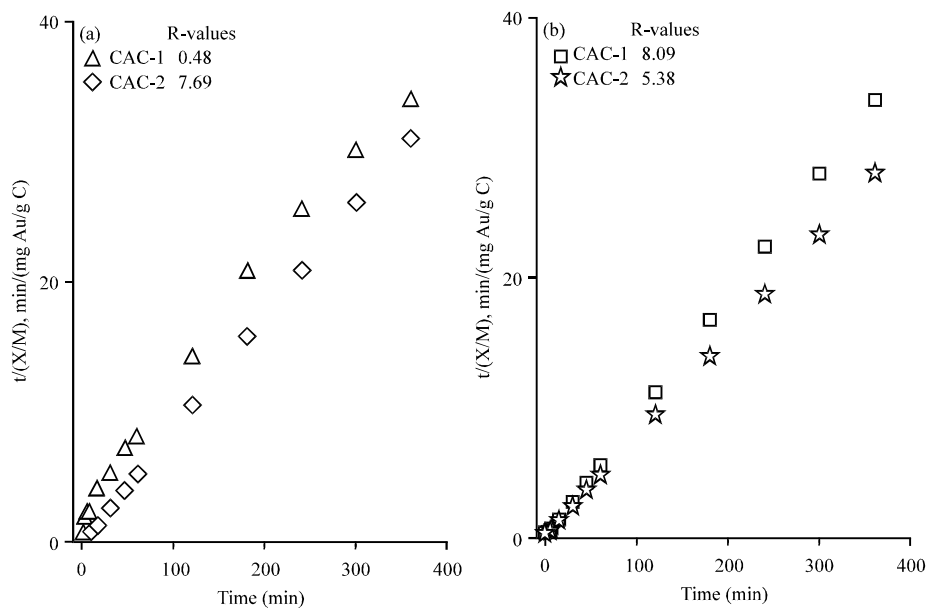


Fig. 4(a-b): Rate of gold adsorption for activated carbons

The effect of particle size on gold adsorption kinetics was studied by some researchers (Cho *et al.*, 1979; Fuerstenau *et al.*, 1987; Le Roux *et al.*, 1991; Van Deventer, 1984) and proposed different kinetic models. To determine rate limiting step and the diffusion rate constant, k_i the following equation was used:

$$k_i = q_t/t^{0.5} \quad (9)$$

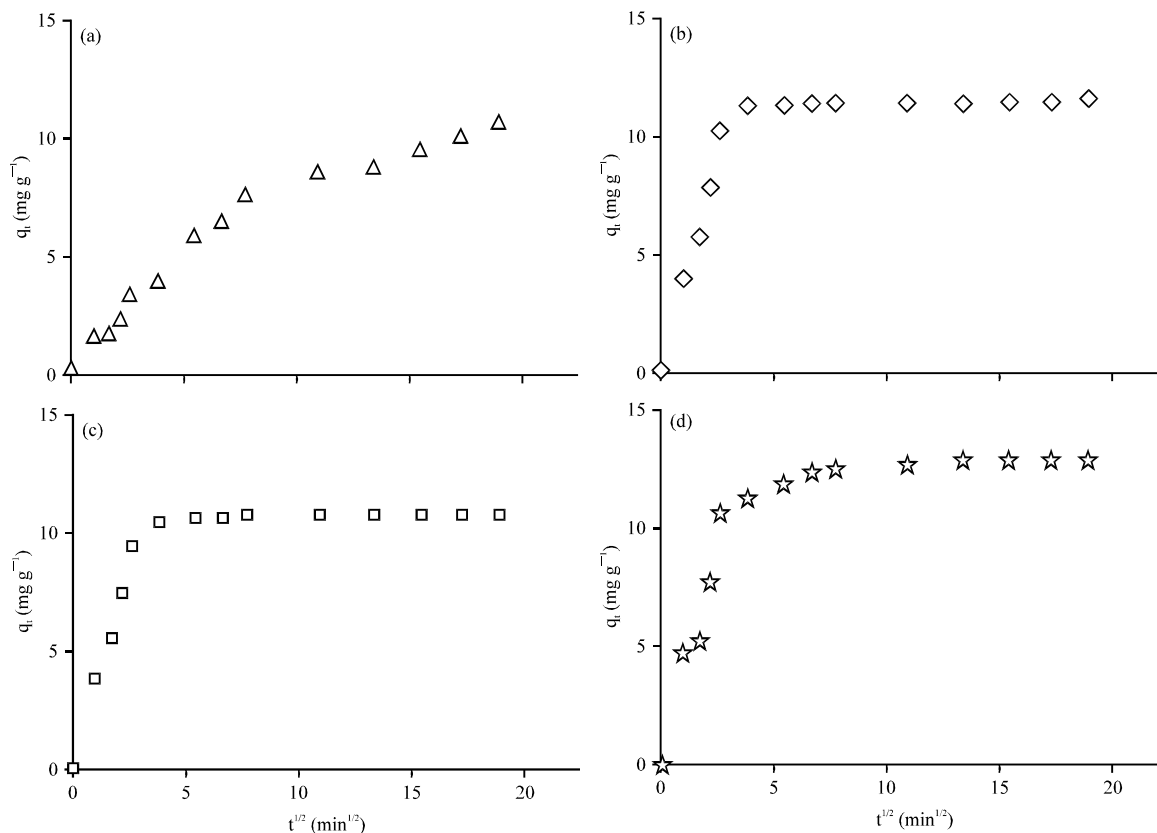


Fig. 5(a-d): Intra-particle diffusion kinetics for adsorption of gold onto the activated carbons, (a) CAC-1, (b) CAC-2, (c) LAC and (d) MAC

The plots of intra-particle diffusion mechanism are shown in Fig. 5. The plots shows multi-linearity (shows three steps adsorption). The first portion was very sharp which was ascribed as diffusion of adsorbate onto the external surface of adsorbent and indicates the fast adsorption rate. It was expected, since interaction between the adsorbent and adsorbed molecules on unit area was increased due to the small particle size of CAC-1, LAC and MAC. On the other hand, the sharper portion was not seen for CAC-1 due to the big particle size. The boundary layer thicknesses (C) also supported this result. When the boundary layer thicknesses was evaluated, the values were close with each other and it showed that the accumulation rates of gold onto surface of all the adsorbents were same except CAC-1. The boundary layer of CAC-1 was smaller than adsorbents. It could be concluded that the rate of gold adsorption is rapid for activated carbon with small particle size. In plant operation, adsorption rate is one of the most important factor which affects gold loss. Gold loss will be minimizing by rapid adsorption even at low concentrations of carbon in the pulp (Yalcin and Arol, 2002). It can be conclude from above statement, application of magnetic activated carbon instead of granular activated carbon in gold metallurgy will decrease the gold loss.

Gold elution

Carbon loading: In order to investigate the elution, behavior of the activated carbons were loaded with gold. Carbon loading values are presented in Table 2. The results can be concluded nearly all the adsorbents have same carbon loading values. An investigation showed that about

Table 2: Carbon loading on activated carbon

Code	Gold on AC (mg Au/g C)
CAC-1	47.13
CAC-2	47.98
LAC	48.28
MAC	47.13

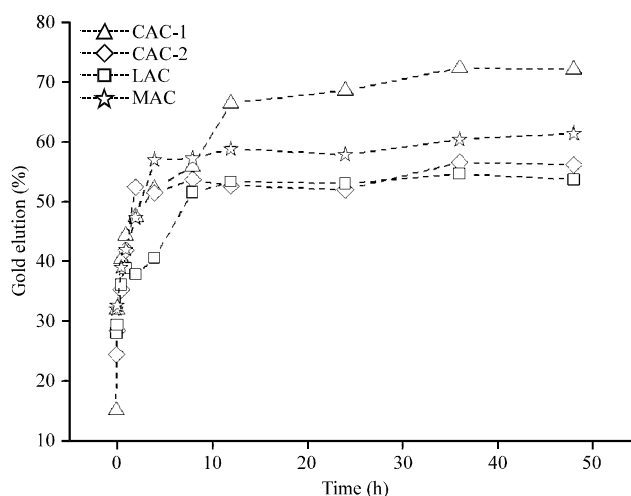


Fig. 6: Rate of elution of activated carbons by Atmospheric Zadra Elution for 48 h

500 Å² per gold complex occupied on surface of activated carbon and less than 1% of the AC surface area is occupied by the gold cyanide ion pair. Therefore, ultimate carbon-loading capacity is independent of particle size.

Atmospheric Zadra elution: The percentage of gold elution depending on time is given in Fig. 6. Like adsorption kinetic rate, it was expected that gold elution rate of CAC-2, LAC and MAC would be faster than that of CAC-1 due to the particle size effect. Since, activated carbon with fine particle size may contact with strip solution faster than the big size depends on the larger external surface area. However, the results indicated that the stripping reaction favored CAC-1 because of coarser carbon particles. The results indicate that CAC-1 had eluted 70% of gold after 48 h, where as CAC-2, LAC and MAC eluted between 50-60%.

The mass transport is the rate limiting step in the elution and occurring in packing area on the activated carbon surfaces, followed by a diffusion process occurring in the meso- and micro-pores within the absorbent structure. Figure 6 indicated that gold elution rate was very fast in the first hour. It could be due to surface reaction (gold on the surface sites of the activated carbons were removed very fast by strip solution). As gold elution percent was compared with particle size of activated carbon, it was clearly seen that gold elution percent of activated carbons for fine particle size was lower than coarser and gold elution percent was inversely dependent on particle size. In the present study, Atmospheric Zadra Elution batch technique were carried out, might not have enough force adsorbed gold locked up in micropores due to the time, pressure, temperature of solution or other effects. However, the effect of activated carbon particle size on the gold elution is beyond the scope of the present study but our investigation is still ongoing on.

Table 3: Gold elution percentage by filter press method

Code	Gold elution (%)	
	Davidson solution	AARL solution
CAC-1	64.6	62.1
CAC-2	93.2	89.4
LAC	91.4	90.8
MAC	89.1	92.3

Filter press elution: Results of gold recovery with Davidson solution and AARL solution in filter press for CAC, LAC and MAC are shown in Table 3. By comparing gold recovery of CAC-1 and CAC-2, it was noted that particle size affected the gold recovery which increased with decreasing the particle size as expected. Besides, it was found that the percentage of gold recovery from LAC and MAC carbon had the nearly same as that from the commercial activated carbon (Elnathan, 2007). It indicated that gold cyanide which was adsorbed on the activated carbon was quickly recovered from the activated carbon by filter press method using Davidson and AARL solution and these methods were faster and more efficient than the Atmospheric Zadra Elution process.

CONCLUSION

In the light of the experimental results, the following conclusions were obtained:

- The activated carbons produced from Tunçbilek lignite had a remarkable BET surface area which was primarily contributed by micropores. The average pore diameters were between 2.28 and 2.44 nm, indicative of its micropores character. Beside, the comparison of adsorption isotherms, surface area and porosity values indicated that surface area, micro and mezzo-pore area of MAC had lower than LAC. This could be attributed to modification process in which Fe_3O_4 was adsorbed by activated carbon
- The adsorption isotherm (K-values) of all the activated carbons is well in the range of K-values of CAC. Modification process did not significantly affect the adsorption capacity. As a result, it was found that the activated carbons (LAC and MAC) prepared from Tunçbilek lignite had good loading capacities. In addition, by comparing the K-value of CAC-1 and that of CAC-2, it could be seen that K-values increased while decreasing the particle size of activated carbon as expected
- The percentage of gold adsorption onto CAC-2, LAC and MAC showed the same trend, they adsorbed more than 80% of gold in the first 15 min and nearly 100% next 45 min. On the other hand, the coarser carbon, CAC-1, adsorbed nearly 30% of gold first 15 min and 65% next 45 min due to the particle size effect
- To understand the adsorption mechanism depending on the particle size, intra-particle diffusion model was also used. It could be said that the rate of gold adsorption is rapid for activated carbon with small particle size. In plant operation, adsorption rate is one of the most important factor which affects gold loss. Application of magnetic activated carbon instead of granular activated carbon in gold metallurgy will decrease gold loss
- In order to investigate the elution behavior of the activated carbons, activated carbons were loaded with gold. Nearly same carbon loading values for all activated carbons were obtained. These data showed that the ultimate carbon-loading capacity was virtually independent of particle size and activated carbon types which were used in the present study

- The elution characteristics of the activated carbons were investigated by Atmospheric Zadra methods and Filter Press methods. In Atmospheric Zadra methods, gold elution rate was very fast in the first hour, so it could be said that elution might be controlled by surface reaction and gold on the surface sites of the activated carbons were removed very fast by strip solution. Experimental results also showed that gold elution percent of activated carbons with fine particle size was lower than coarser and gold elution percent was inversely dependent on particle size and the stripping reaction favored CAC-1
- Results of gold recovery with Davidson solution and AARL solution in filter press indicated that particle size affected the gold recovery which increased with decreasing the particle size as expected. Besides, the percents of gold recovery from LAC and MAC carbon had the nearly same as that from the commercial activated carbon. It was also concluded that filter press method using Davidson and AARL solution were faster and more efficient than the Atmospheric Zadra Elution process
- In summary, this study demonstrates that the active carbon produced from Tunçbilek lignite and modification form can be employed in gold metallurgy. Especially, magnetic activated carbon, with high adsorption and desorption capacity which should offer an easy separation of gold loaded activated carbon from slurry may be a promising sorbent for gold metallurgy. However further work is necessary to understand the system and to obtain a better perspective about applicability of magnetic activated carbon in plant operation

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