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Recovery of Activated Carbon Powder from Aqueous Solution in the Flotation Method By Using Pine Oil

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Abstract: *In this study, the authors have assessed a number of factors that effect the ability of activated carbon recovery by flotation, such as chemical foaming, concentration, air flow rate and flotation time. The results show that pine oils have the good ability to inform foaming (smooth and durable) and suitable for recovery of activated carbon powderin the flotation method. In addition, the combination of pine oil and CH_3COOH with volume ratio 1:1 can achieve excellent ability of recovery activated carbon powder(98%) within 10 minutes.*

Keywords: *recovery, flotation, activated carbon, pine oil, acetic acid*

1. INTRODUCTION

In water treatment, what attracted scientists to small-size (μm)absorbing substances (particles) was their typical physical and chemical properties.The smaller absorbing substances are, the larger contact surface is and the more effective the absorbing capacity of target substances is (metal ions, suspended solids in solution). It is difficult to remove or recover nonmagnetic aborbing sustances but magnetic ones after water treatment. So far, many methods of nonmagnetic small-size aborbing sustance separation have been developed such as co-precipitation method, filtration method, sedimentation method and flotation method, of which flotation method can separate small-sized colloidal particles and suspended solids from a solution most effectively, easily and cheaply[1-2].

For flotation method, target substances will be water-repellent or have water-repellent functional groups. If such substances are water-retaining or have water-retaining functional groups, it is essential to add surface-active agents or foaming aids such as sodium dodecyl essential sulfate (SDS) [3], dodecanamine (C12H27N) [4], aluminum sulfate Al2(SO4)3 [5], etc. Nevetheless, it is worthly to give more attention to the residual compounds in the solution after water treatment due to its high toxicity to human health and surroundings.Therefore, the matter of great importance is continuation of finding out eco-friendly flotation foaming chemicals with its high treatmentcapacity.

γ –PGA is a foaming chemical that we have researched and proven its foaming capacity [7]. γ –PGA,which could be used as an ingredient of “Natto”– one of traditional Japanese food, has been used in cosmetic and food industries, etc; thus, it is completely non-toxic to humans and the environment[8-11]. However, its price is rather expensive since it is imported from Japan. Therefore, it is becoming almost essential to continue finding out eco-friendly foaming chemicals made in Vietnam with its high treatmentcapacity.

Pine oil is distilled that uses steam from needle leaves, young stems and cones of some pines such as Pinus sylvestris. Chemically, pine oil mainly consits of Aromatic terpene alcohols. It also may contain hydrocarbons, ethers and esters terpenes.Its specific components depend on various factors such as type of pine or input materials (needle leaves, young stems and cones). Moreover, pine oil is relatively low toxic to human. In addition, recovery of activated carbon powder in the flotation method by using pine oil has not been reported in Vietnam and in foreign countries. On this topic, we have studied the recovery of activated carbon powder by using pine oil as a foaming agent and obtained high amount of activated carbon within a short time.

In flotation method, ethanol is used as a traditional foaming agent [6] in order to reduce surface tension of water and to generate more gas bubbles. In our research, we have discovered that foaming capacity of acetic acid was equal to that of ethanol. In addition, the combination of pine oil and acetic acidsignificantly reduces added chemicals and improve ability of recovery of activated carbon powder from aqueous solution.

2. EXPERIMENT

2.1. Objectives of the research

Assessing factors that effect the ability of activated carbon powder (< 50µm) recovery by flotation using pine oil through the below experiment: comparing foaming capacity and ability of activated carbon recovery of the combination of pine oil and γ–PGA to the combination of pine oil and CH_3COOH (two chemicals have been assessed in the previous researches); effect of content of foaming chemical, flow of input gas and flotation time on the ability of activated carbon recovery.

2.2. Chemicals, instrument and equipment for research

2.2.1. Chemicals

- Gamma-polyglutamic acid (γ–PGA): Nippon Polyglu Company – Japan

- Acetic acid (CH3COOH), China

- Pine oil, Vietnam

-Activated Carbon, Wako Chemical Company, Osaka, Japan

- Distilled water.

2.2.2. Instrument and equipment

Fig1. *Flotation tube*

- Flotation tube (h =60 cm, D = 4cm, Fig.1)
- Input gas Flow Meter
- Aerator
- Glass cups 100ml, 250ml.
- Volume flasks 100ml,250ml, 500ml.
- Glass pipettes: 2ml, 3ml, 5ml, 10ml.
- Plastic pipettes: 0,5ml, 1ml
- Filter paper
- MLSS SS-10F Suspended substances concentration gauge (Japan).
- Eutech Instruments Turbidimeter Tn-100 (Japan), (Fig.2).

SS-10F

MODEL No.	SS-10F
Measurement Method	Near-infrared pulses transmitted light
Measurement Range	0~30000 mg L^{-1}
Sample Volume Required	30 ml
Accuracy	10 mg L^{-1}(0~10000 mg L^{-1}) 100 mg L^{-1}(10000~30000 mg L^{-1})

TN-100

Fig2. *MLSS SS-10F Suspended substances concentration gauge and Eutech Instruments Turbidimeter TN-100*

MODEL No.	TN-100
Measurement Method	ISO 7027 compliant nephelometric method (90°)
Light Source	Infrared-emitting diode (850 nm wavelength)
Measurement Range	0 - 1000 NTU
Sample Volume Required	10 ml
Accuracy	±2% of reading for 0 to 500 NTU; ±3% of reading for 501 to 1000 NTU.

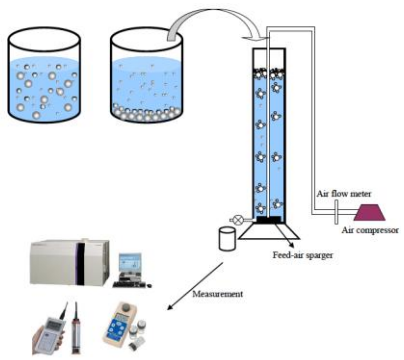

Fig3. *Flotation model*

2.3. Experiment

2.3.1. Operation principles: flotation method was performed by aerating air in aqueous solutionwith activated carbon powder (<50 µm) and foaming chemical. During aeration, activated carbon were mixed with generated air bubbles, the bubbles floated to the surface. We removed activated carbon from the solution by scumming in the surface (Fig.3).

2.3.2. Experiment preparing activated carbon powder [7]: Pour 25 gram of activated carbon into 10 liters of distilled water, and stir to form a solution contained 0.25% of activated carbon (solution A). Settle within 3 hours in order to recover large-size activated carbon in the bottom of the water tankand solution in the surface was used in the experiment assessing flotation method in activated carbon powder recovery.Size of activated carbon particles was measured depending on sedimentary time, and after 3 hours of sedimentary, diameter of almost residual carbon partiles in the solution was under 50µm (solution B), (Fig. 4,5).

Sedimentary rate (u_t: m s^{-1}) was presented in the following equation:

$$u_t = \frac{D^2 g(\rho_p - p)}{18\mu}$$

Of which:

D: diameter of solid particles [m]

µ : ropiness of liquid [kg m-1 s-1]

g : gravitational acceleration [m s-2]

ρ : concentration of solution [kg m-3]

ρp : concentrationof solid particles [kg m-3]

Table1. *The relationship between sedimentation rate and particle size*

Diameter [μm]	u_t [cm h^{-1}]
200	78
150	44
100	20
75	11
50	5
25	1
10	0.2
1	0.002
0.1	0.00002
0.01	0.0000002
0.001	0.000000002

(a) Before **(b) After**

Fig4. ***Activated carbon solution (a) before and (b) after 3 hours of sedimentary***

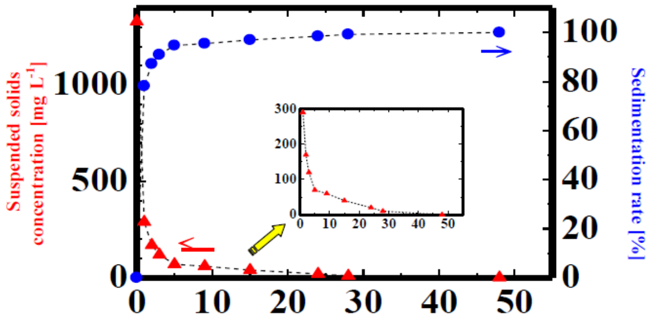

Fig5. *Concentration and sedimentary rate of activated carbon in aqueous solution*

2.3.3. Experiment assessing factors that effect flotation

Effect of foaming chemicals on activated carbon recovery In this experiment, we have assessed 3 foaming chemicals: γ - poly glutamic acid (γ -PGA), CH_3COOH, and pine oil.

- Pour 300ml of solution B (concentration of activated carbon: 300mg/ L) into the flotation tube, after that add γ–PGA or CH_3COOH or pine oil as foaming chemicals into such solution (assessing each chemical separately).

- Aerate air in the tube within 30 minutes with its flow of 3 L/min.

- Recover water in the tube by opening the valve in the bottom of the tube.

- Obtained solution was measured concentration of activated carbon, whereby the ability of activated carbon recovery by using the chemicals was determined by the below formula:

$$C(\%) = \frac{(C_t - C_s)}{C_t} * 100$$

Of which:

- C:ability of activated carbon recovery (%).

- C_t: concentration of activated carbon put into solution prior to flotation (mg L^{-1}).

- C_s: concentration of activated carbon in solution after flotation (mg L^{-1}).

3. Results and Discussion

3.1. Effect of foaming chemicals on the ability of activated carbon recovery

Conditions of experiment: concentration of each chemical: 0.05%; flotation time: 30 minutes; air flow rate: 3 L/min; concentration of initial activated carbon: 300 mg L^{-1}[7].

The research results were presented in the table 2. Visual and mechanical measurement showed that the ability of activated carbon recovery of CH_3COOH(65.5%) ,γ –PGA (68.0 %) and pine oil (68.4 %) are nearly equal to each other.

Table2. *Effect of foaming chemicals on processing efficiency*

Chemical	Processing efficiency[%]	± s.d.
CH_3COOH	65.5	± 1.5
γ –PGA	68.0	± 0.4
Pine oil	68.4	± 1.6

(*s.d.: standard deviation*)

Since surface tension of CH3COOH is smaller than that of water, generated air bubbles are much more smaller when aerating air in a solution with such chemical than when aerating air in a solution without such chemical. These air bubbles pushed activated carbon up the surface and processing efficiency achieved 65.5%. Surface tension of CH_3COOH and water at 20^{o}C were 27.6 and 72.7 mN/m, respectively. The ability of activated carbon recovery was significantly improved when concentration of CH_3COOH increased from 0.05% to 0.1%, 0.5% and 1%.

In this research, we have found out a new foaming chemical, pine oil. The ability of activated carbon recovery using pine oil was equal to that using γ –PGA [7] andCH_3COOH. Surface tension of pine oil has not reported in materials up to now. Nevertheless, according to the experiment, its ability of activated carbon recovery was excellent (68.4%) because quantity of generated bubbles when using this oil was large and small- size.

In addition, according to the previous research [7], the foaming capacity of the combination of CH_3COOH and γ –PGA was much more excellent than that of one separate chemical. Therefore, in our research, we had combined CH_3COOH and pine oil, and the result was remarkably improved. Generated air bubbles were very small, and rate of pushing activated carbon up the surface was much higher. It was observed that pine oil had not only the capacity of small foaming but also the capacity of bonding activated carbon particles together whereby its processing capacity was better than γ –PGA's.

(A) (B)

Fig.7. *Solution with chemical (A) CH_3COOH&(B) pine oil before (left) and after (right) flotation*

Fig8. *Solution with the combination of CH3COOH and pine oil before (left) and after (right) flotation*

3.2. Effect of Concentration of Chemicals on the Ability of Activated Carbon Recovery

3.2.1. Effect of concentration of CH3COOH on the ability of activated carbon recovery

When concetration of CH_3COOH was increased from 0.005% to 1%, the result was presented in the Chart 1. According to the Chart 1, concentration of CH_3COOHgreatly impacted on the ability of activated carbon recovery. This could be explained that when concentrtion of CH_3COOH increased, the higher quantity of generated air bubbles was, the larger contact surface of activated carbon with the bubbles was, resulting in improved processing capacity. This trend has also been demonstrated in the previous articles [7-10].

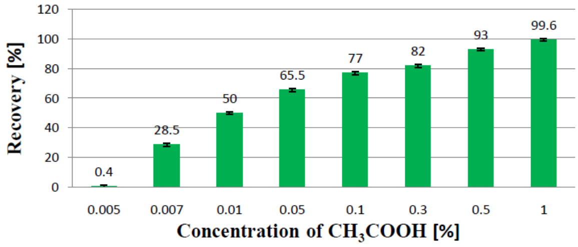

Chart1. *Effect of concentration of CH_3COOH on the abilityof activated carbon recovery*

Conditions of the experiment:

- Foaming chemicalwas CH_3COOH
- Flotation time was 30 minutes
- Air flow rate was 3 $Lmin^{-1}$
- Concentration of initial activated carbon was 300 mgL^{-1}

3.2.2. Effect of concentration of pine oil on the ability of activated carbon recovery

When concetration of pine oil was increasedfrom 0.005 % to 0.5 %, the result was presented in the Chart 2. According to the Chart 2, concentration of pine oil greatly impacted on the ability of activated carbon recovery.The higher concentration of pine oil was, the higher quantity of air bubbles was. We have not *presented* the experiment result using 1% of pine oil because quantity of air bubbles in this case was very high and the bubbles were rapidly poured out the tube within 5 minutes. The ability of activated carbon recovery was improved due to the foaming capacity when increasing concentration of pine oil. 0.5% of pine oil can achieve excellent ability of recovery activated carbon (99%) within 30 minutes.

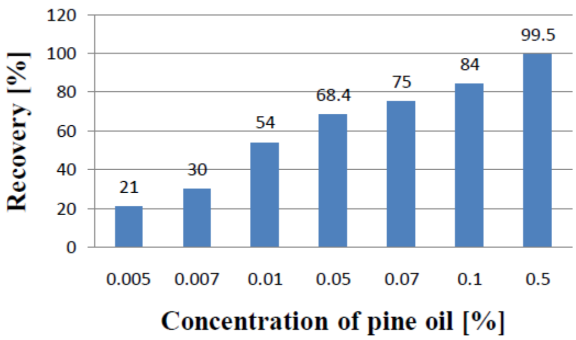

Chart2. *Effect of concentration of pine oil on the abilityof activated carbon recovery*

- Foaming chemical waspine oil
- Flotation time was 30 minutes
- Air flow rate was 3 $Lmin^{-1}$
- Concentration of initial activated carbon was 300 mgL^{-1}

3.2.3. Results of activated carbon recovery of the combination of pine oil and acetic acid

In this experiment, we have assessed the effect of the combination of pine oil and acetic acid on the ability of activated carbon recovery. Particularly, we changed volume ratio and concentrtion of two chemicals as follow: pine oil : acetic acid = 1:0 (0.05 % of pine oil, without axit acetic), 1:0.1 (0.05 % of pine oil and 0.005 % of acetic acid), 1:0.3 (0.05 % of pine oiland 0.015 % of acetic acid), 1:0.5

(0.05 % of pine oil and 0.025 % of acetic acid), 1:1 (0.05 % of pine oiland 0.05 % of acetic acid), 0.5:1 (0.025 % of pine oil and 0.05 % of acetic acid), 0.3:1 (0.015 % of pine oiland 0.05% of acetic acid), 0.1:1 (0.005 % of pine oiland 0.05 % of acetic acid), 0:1 (without pine oil, 0.05 % of acetic acid).

The experiment result was presented in the Chart 3. One chemical (pine oil or acetic acid) could achieve ability of activated carbon recovery (70%), and the combination of pine oil and CH3COOH could improve such ability, and such combination with volume ratio 1:1 could achieved the most excellent ability. After this experiment, we assessed other factors that effect the ability of activated carbon recovery using the combination of pine oil and acetic acid with volume ratio 1:1.

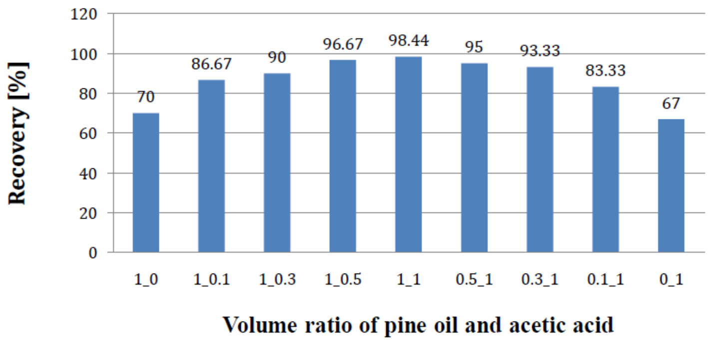

Chart3. *Ability of activated carbon recovery when changing volume ratio of pine oil and acetic acid*

- Foaming chemical waspine oil and acetic acid
- Flotation time was 30 minutes
- Air flow rate was 3 $Lmin^{-1}$
- Concentration of initial activated carbon was 300 mgL^{-1}

3.3. Effect of airflow rate on the ability of activated carbon recovery

Experiment assessing effect of air flow rate on the ability of activated carbon recovery was carried out and its results were presented in the Chart 4.The results showed that the higher air flow rate was, the more excellent ability of activated carbon recovery was. It meaned that when air flow rate was increased, quantity of bubbles in the solution would be increased, resulting in increasing rate of pushing activated carbon particles up the surface. In this research, we have presented the experiment with air flow rate of 4 L/min. because the bubbles were rapidly poured out the tube when this flow was higher than 4 L/min., then the result was not exact. This trend has also been demonstrated in the previous articles [12 -13].

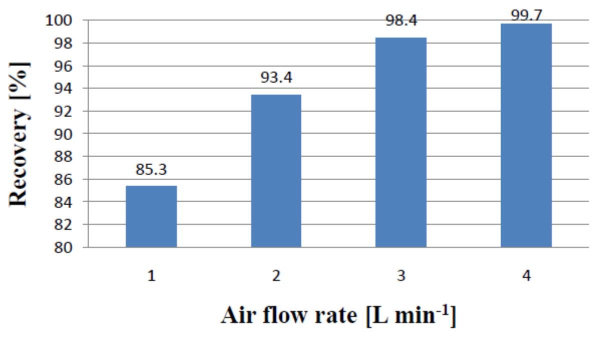

Chart4. *Effect of air flow rate on the ability of activated carbon recovery*

Conditions of experiment:

- Flotation chemical:combination of pine oil or acetic acid with volume ratio 1:1.
- Flotation time: 30 minutes.
- Concentration of initial activated carbon: 300 mg/L

3.4. Effect of flotation time on the ability of activated carbon recovery

After assessing effect of chemical, concentration, air flow rate, we have continued assessing the effect of flotation time on the ability of activated carbon recovery under the following conditions: flotation chemical was the combination of pine oil or acetic acid with volume ratio 1:1, air flow rate was 3 L/min., and concentration of initial activated carbon was 300 mg / L. Its results were presented in the Chart 5. The results showned that the longer flotation time was, the more excellent ability of activated carbon recovery was within 30 minutes. After the 30^{th} minute, the ability of recovery would be lightly reduced since a part of activated carbon was broken after bind with pine oil and aerated too long. The rate of flotation rate by using pine oil were much better than that by using γ –PGA[7] as a flotation chemical. Therefore, the optimal flotation time was 10 – 30 minutes.

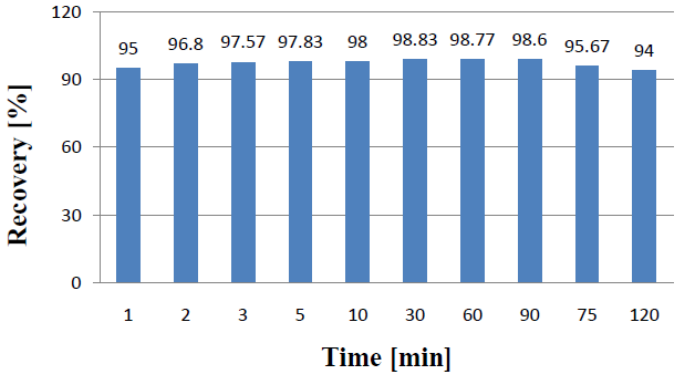

Chart5. *Effect of flotation time on the ability of activated carbon recovery*

Conditions of experiment:

- Flotation chemical: pine oil or acetic acid
- Flotation time: 30 minutes
- Airflow rate: 3 L/min
- Concentration of initial activated carbon: 300 mg / L

4. Conclusions

Though the research, we have assessed some factors that effect the ability of activated carbon recovery in flotation method by using pine oil. The results showed that the ability of activated carbon recovery by using pine oil was nearly equal to that by using γ –PGA andCH_3COOH. However, the binding capacity of activated carbon particles with air bubbles by using solution with pine oil was the best. In addition, the combination of CH_3COOH and pine oil with the volume ratio 1:1 could achieve the ability of activated carbon recovery (98%) within 10 minutes. This method will be applied to recover heavy metal ions in aqueous solution by indirect recovery of activated carbonabsorbingheavy metal ions, and a large-scale flotation model will be developed for putting into practice in the future.

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