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CHARACTERIZATION AND PERFORMANCE EVALUATION OF REUSED WABE AS FLOCCULANTS AGENT TO TREAT INDUSTRIAL PAPER MILL WASTEWATER

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Abstract

The present research mainly to explore the performance of customize flocculants agent made by reusable waste (WABE) to treat actual wastewater from different industry. The effect of mixing rate (10, 20, 30, 150, 175 and 200 rpm), sedimentation time (5, 15 and 30 min), initial pH (5, 6, 7 and original pH) of initial wastewater and flocculation dosage (2, 5 and 10% v/v) were investigated based on turbidity removal. WABE characterized by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Fourier Transform Infrared Spectroscopy (FTIR). The result shows flocculants agents have high turbidity removal up to 90% at optimum condition. This study also provides strong evident WABE can be developed as flocculants agent at low cost and low maintenance.

Keywords: Flocculation, Wastewater, Flocculants.

1. Introduction

Flocculation often used in wastewater treatment. Traditional wastewater treatment involve three stages process beginning with a primary solid separation to removed suspended solids followed by a secondary biological treatment system to reduce biochemical oxygen demand (BOD) and inorganic dissolved nutrient [1]. The third stage was biological treatment to nutrient reduction and possible disinfection [1]. It is well known that solid-liquid separation by flocculation is an important process for waste water treatment, sludge dewatering, and pulp and paper production, as well as in pharmaceutical, cosmetic, and metal working industries [2]. Generally, the floc-

Abbreviations

BOD	Biochemical oxygen demand
CET	Cellulose extraction Timble
CPO	Crude palm oil
FTIR	Fourier Transform Infrared Spectroscopy
GHG	Greenhouse gas
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
SBE	Spent bleaching earth
WABE	Waste Activated Bleaching Earth

culants used in water treatment can be classified into three groups: inorganic flocculants (alum, ferrite flocculants, or poly-aluminium chloride), synthetic organic flocculants (polyacrylamide derivatives), and naturally occurring flocculants (sodium alginate or microbial flocculants [3]. There are different types of mechanism that initiate flocculation process. Flocculation occurs either by bridging, charge neutralization, polymer complex formation, and depletion flocculation or by combination of two or more of this mechanism [4]. Besides, urban industrial activity has long been identified as a major source of contaminants for aquatic environments, via atmospheric deposition and wastewater discharge [5]. The pulp and paper industry is the sixth largest polluter (after oil, cement, leather textile, and steel industries) discharging a variety of gaseous, liquid and solid wastes into the environment [5]. In this study, Waste Activated Bleaching Earth (WABE) used as flocculants agent that can enhance coagulation process and form precipitate. Spent bleaching earth (SBE) derived from the degumming and bleaching of crude palm oil (CPO) from physically refinery palm oil is commonly disposed of at landfills at high cost [6]. The spent bleaching earth (SBE) contains about up to 30% (w/w) of residual oil that rapidly oxidizes to the spontaneous auto-ignition point, and also produces unpleasant odors [7]. Currently in Malaysia, the most common practice is disposal of SBE at landfills which causing fire and pollution hazards due to the degradation of the residual oil in it and the associated greenhouse gas (GHG) emission upon its disposal [6]. WABE consist of elements that can initiate coagulation and flocculation process to occur. In that sense, the element that contain in WABE can be used to treat wastewater. The development of recyclable flocculants could minimize the cost of flocculation and could solve the problem of exhausted flocculants disposal [8]. Therefore, the aim of this study was to investigate the potential of WABE as flocculants agent by treating real wastewater.

2. Materials and Methods

2.1. Sampling of paper mill wastewater

Wastewater collected from paper mill's industry in Kajang, Selangor. The colour of the sample is brown and in non-homogenous liquid form. The sample was kept in freezer for 12 hours before undergo Jar Test.

2.2. Preparation of flocculants agents from WABE

Powder of WABE was de-oiled using Sochlet, Cellulose extraction Timble (CET) and hexane (Bendosen, Norway). Hexane was heated up to 90°C to allowed condensation process to occur and the oil will be collected at the bottom part.

Hexane added continuously until the oil completely removed from the WABE. De-oiled WABE collected and cooled for 30minutes. Saturated sulphuric acid (Friendemann Schmidt, United Kingdom) and sodium hydroxide (Friendemann Schmidt, United Kingdom) were added to the de-oiled WABE and stirred another 30 minutes. 2L distilled water was added while stirring for another 45 minutes. Solid particles will suspend at the bottom and the liquid phase was collected to form acid or base flocculants for further investigation.

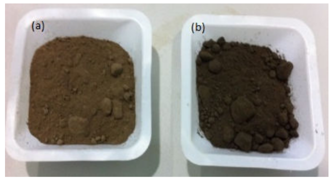

Fig.1. Raw WABE collected from palm oil industry (a) Felda Iffco and (b) Wilmar edible oil.

2.3. Characterization of flocculants agent

2.3.1. ICP-MS and FTIR

Element present in the flocculants were determined by using ICP-MS (Perkin Elmer 9000, USA). Meanwhile, FTIR (Nicolet 6700, USA) analysis was done on flocculants to determine the functional group that might involve during flocculation process .The sample were prepared in 5 separated bottled labelled A, B, C, D and E. Each bottled contained 10 ml of the flocculants sample. The sample prepared in homogenous form.

2.4 Flocculation experiment

The ratio of the combination acid flocculants and basic flocculants is 1:1. The flocculating experiments were conducted at room temperature. Table 1 show mixed flocculants used in Jar test.

Table 1. Mixed flocculants used to treat actual wastewater.

FLOCCULANTS	COMBINATION OF LIQUID FLOCCULANT
A	Acid Wilmar + Base Wilmar
B	Acid Wilmar + Base Felda Iffco
C	Acid felda Iffco + Base felda Iffco
D	Acid Felda Iffco + Base Wilmar
E	Combination of flocculant A,B, C and D

Jar test were conducted to study the effect of flocculants dosage (2, 5 and 10%), mixing rate (10, 20, 30, 150, 175 and 200 rpm), sedimentation time (5, 15 and 30 min), initial pH of wastewater (5,6 and 7), and type of flocculants (A, B, C, D and E). First treatment was to determine the best mixing rate for flocculation followed by sedimentation time, initial pH of wastewater, flocculants dosage and types of flocculants. For each treatment 10ml of supernatant were collected for turbidity test. The treatments were repeated for three times.

3. Results and Discussion

3.1. ICP-MS analysis

The element from each flocculants labelled A, B, C, D and E were determined by using Inductively Coupled Plasma Mass Spectrometry (ICPMS) from Chemical Analysis Laboratory at Faculty Science and Technology. Table 2 shows ions present in the flocculants. The aim for this element determination is to get an overview which element responsible to initiate flocculation process. Besides, based on the commercialize flocculants agents and previous research, there might be similarities in element composition that being used. The ions selected might have an impact for flocculation process as reported from the previous study. For example, the colloid surface charge decreases due to the charge neutralization mechanism, enhancing the destabilization of the colloidal particles in water [9]. In addition, the increase in the turbidity and COD removal efficiency at pH 5-8 to hydroxyl ions reacting with Fe species to produce more ferrite polymers can improve the bridging flocculation [10].

Table 2. Elements constituent in flocculants.

Flocculants Ions	A (ppm)	B (ppm)	C (ppm)	D (ppm)	E (ppm)
Mg	66.68	60.87	71.90	54.05	51.15
Al	1.30	1.29	31.57	45.17	5.06
Mn	0.97	7.30	9.57	12.23	7.50
Fe	2.40	8.15	32.13	50.57	13.80
Co	0.02	0.07	0.08	0.06	0.07
Ni	0.06	0.99	0.10	0.10	0.10
Cu	0.01	0.11	0.28	0.21	0.06
Zn	0.01	0.16	0.46	0.32	0.07
Rb	0.06	0.09	0.08	0.05	0.05
Cd	-	0.03	0.07	0.08	-
Cs	-	-	-	-	-

Furthermore, in aqueous solution, the trivalent Fe(III) ions readily undergo hydrolysis, complexation / polymerization and precipitation [11]. Ions Flocculants ions such as Mg2+, Al3+ and Fe2+/3+ can be a major role in flocculation. Besides, in previous study, an ion present in the flocculants can trigger flocculation process.

3.2. FTIR analysis

The functional group from flocculants A, B, C, D and E were determined using Fourier Transform Infrared Spectroscopy (FTIR) from Chemical Laboratory at Faculty of Engineering. Figure 2 shows FTIR graph spectrum analysis for each flocculants. The functional groups present in flocculants as Table 3. Previous research had noted that certain functional groups are responsible for the formation of flocs.

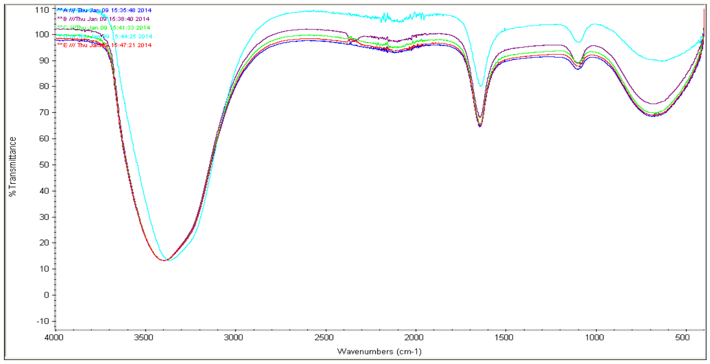

Fig.2. FTIR graph spectrum for each flocculants.

Table 3. Functional groups presence in flocculants.

Subject Functional Groups	Wavelength 698reading (cm^{-1})	A	B	C	D	E
Amine	3300-3500	/	/	/	/	/
Alkyne	2100-2300			/		
Alkene	1640-1680	/	/	/	/	/
Salfone	1300-1350 1100-1150	/	/	/	/	/
Alkyl chloride	580-780	/	/	/	/	/

There are functional groups that used to initiate flocculation in chemical reaction between flocculants particles and wastewater. Amine group presence can form precipitate or complex compound in chemical reaction involved. The FTIR spectrum of flocculants TJ-F1 shows that it contains carboxyl, hydroxyl and amino groups as well as hydrogen bonds which are the preferred groups for the flocculation process [12]. Besides, Functional group carboxyl, hydroxyl and amino groups as well as hydrogen bonds can act as a key role in bridging the suspended particles to form big flocs during flocculation process [13]. The strong absorbability between the functional groups could make the new produced sludge flocs more compact than the original ones [14]. The functional groups in this experiment give a preliminary data for further research about the mechanism involve in this study.

3.3. Effects of mixing rate

Figure 3 showed under slow rate mixing condition, the percentage of turbidity removal was higher compared to rapid mixing rate. The range of percentage turbidity removal under slow mixing rate was 87.19-90.64%.

The flocculants used were in ions form, which were stable from the beginning of flocculation process. The highest average of percentage turbidity removal was at 20 rpm which is 90.07%. Based on the highest average of percentage turbidity removal, flocculants D performs the best compared to other flocculants which is 89.00%. In the preliminary study it shows that at higher speed from 150 rpm-200 rpm will disturb the formation and colloidal particles and show high turbidity reading at the end of treatment. In theory, rapid mixing used to disperse the flocculants in surrounding liquid so that the flocculants can react to the entire liquid surrounding uniformly. Besides, increasing the shear intensity increases the number of collisions between aggregates where newly formed aggregate not only break up more frequently but are also limited to a smaller overall aggregate size no matter how much extra flocculants is added [15]. At mixing rate of 200 rpm, lowest percentage of turbidity removal is obtained (84.12%), meanwhile at mixing rate of 20 rpm show highest percentage of turbidity removal at 90.64%.

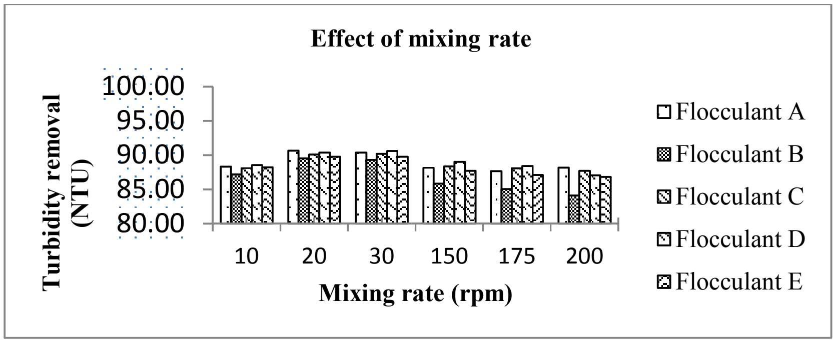

Fig.3. Flocculation at different mixing rate condition.

3.4. Effect of sedimentation time

Sedimentation time used was 5, 15 and 30 min as in Figure 4. The shorter the time of sedimentation shows better flocculation process has occurs.

The times for ionic interaction in the elements present to form flocs can be determines by the time of sedimentation. It is also show how many time required to form stable flocs to settle down. The flocculation performance parameters are settling rate, residual turbidity, sediment density and sediment rheology used to characterize the quality or extent of flocculation achieved with a given flocculants [16]. Its show the flocculation process occurs at rapidly once the flocculants was added. That is an advantage of liquid flocculants where the flocculation process can occur faster. The dispersion of flocculants particles into the surrounding liquid is faster compared to solid flocculants. Besides, the initial stage of flocculation may progress through a fine capture process, forming small, slow settling aggregates [17].

3.5. Effects of initial pH of wastewater

Figure 5 shows effect of initial pH of wastewater. Most of the wastewater pilot plant treatments in Malaysia are using biological treatment to degrade organic solid particles by using microorganism such as bacteria.

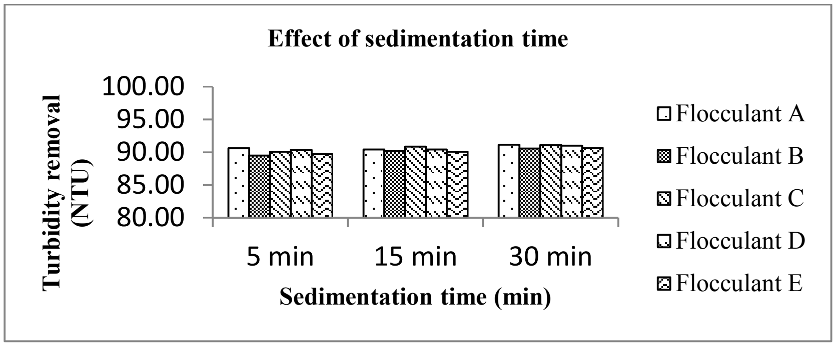

Fig.4. Turbidity reading at different sedimentation time.

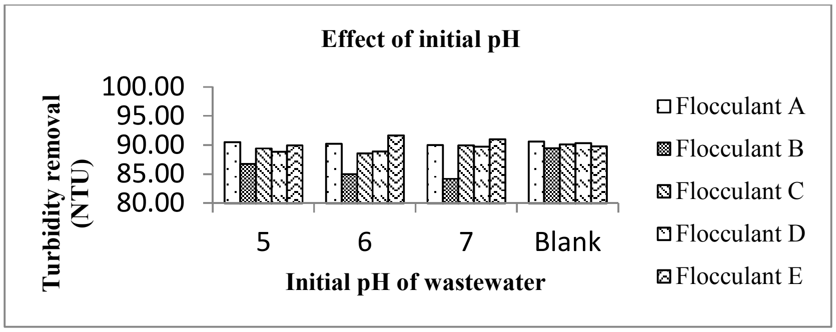

Fig.5. Flocculation at different initial pH of wastewater.

Most bacteria can live near pH neutral to maintain their optimum growth. Thus, we are trying to study the effect of initial pH of wastewater by adding 1M of hydrochloric acid HCl or 1M sodium hydroxide NaoH so that the adjusted to pH 5, 6 and 7. The pH of wastewater was adjusted for desired pH using 1M H2SO4 and 1M NaOH. The original pH of the wastewater is 6.75 and labelled blank. At pH blank, highest turbidity removal were recorded for flocculants A, B, C and D. At pH blank, flocculants E shows slightly lower performance compared at pH 5, 6 and 7. The highest percentage turbidity removal was 91.64% at pH 6 using flocculants E. Previous research have recorded that Aluminium sulfate (AS) has an optimum pH range 4.0 to 7.0 where the optimal coagulation with AS take place at pH values near 5 and 7 [18]. Besides, the optimal flocculating activities for Ca2+, Fe3+, and Al3+ were near neutral pH range 7.0, 6.4 and 7.1 for Ca2+, Fe3+, and Al3+ respectively [19]. These ions were present in the flocculants that we used. Thus, it indicates that the flocculation process might be initiated by these ions present.

3.6. Effects of flocculants dosage

Figure 6 shows effect of flocculants dosage. Flocculants dosages have become a major concern in industrial wastewater treatment especially those company that used bio-flocculants. Flocculating activity and cultivation cost are always the major limitation of bio-flocculants application [20-23].

This study would give another alternative by using reusable activated bleaching earth as flocculants agent. Flocculation behavior is strongly dependent on the amount of polyelectrolyte added and flocculation time [24]. It shows that high flocculants dosage used does not mean it have better flocculation performance. Besides, liquid flocculants used have greater effect on this wastewater in initiating flocculation process. The effect of flocculants dosage 2, 5 and 10% were not very different in flocculation performance based on percentage turbidity removal. The highest turbidity removal was at 10% flocculants dosage by using flocculants A. Flocculation process can occur at low level flocculants dosage due to rapid dispersion of flocculants used in the wastewater.

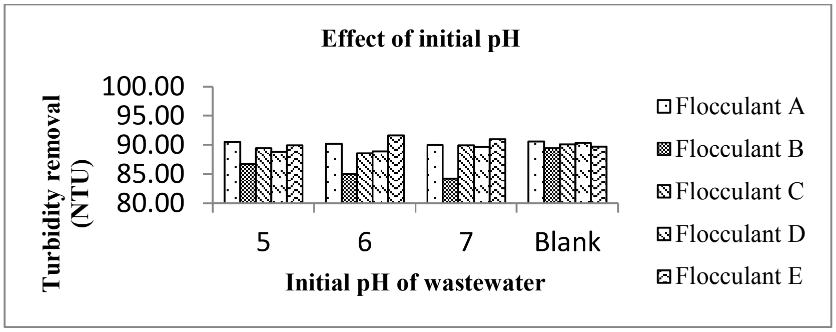

Fig.6. Flocculation at different flocculation dosage.

3.7. Comparison on types of flocculants used

The present of certain element such as aluminium, sulfur, iron is significant to show the performance of the flocculation process. Generally, the flocculants used in water treatment can be classified into three groups: inorganic flocculants, such as alum, ferrite flocculants, or poly-aluminium chloride; synthetic organic flocculants, such as polyacrylamide derivatives; and naturally occurring flocculants, such as sodium alginate or microbial flocculants [3]. In this research, newly customize flocculants were prepared to treat wastewater. The flocculants were labelled A, B, C, D and E had different ions composition. All of the flocculants used contained ions Fe2+, Al3+ and Mg2+ which can initiate flocculation. Besides, there are past research such as flocculants TJ-F1 shows a crystal-linear structure, which can assure more adsorption point to be operative and bridge more particles to form flocs [13]. In addition, at the beginning of flocculation process, different segments (PAM flocculants branches or the main backbone) of the same polymer chain can bind more than one particle very rapidly [25]. Therefore, different flocculants consist of different ions composition where also lead to different flocculation mechanism.

4. Conclusions

The results obtained show customize flocculants from reusable WABE have the ability to initiate flocculation process. The physical and chemical characteristics of flocculants provide strong evidence to support the hypothesis. Element such as iron and aluminium contain have a major function in flocculation process. These researched provide strong evidence that the WABE can perform as flocculants agent at certain condition (low mixing rate, low sedimentation time, almost neutral condition and low flocculants dosage). In this case flocculants D (Mg2+:54.05 ppm; Al3+:45.17 ppm; Fe2+:50.57 ppm) perform the best. These researched provide preliminary data for further enhancement of WABE as flocculants agents.

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