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Microwave-assisted synthesis and characterization of polyacrylamide grafted cellulose derived from waste newspaper for surface water treatment

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ABSTRACT

In this study, polyacrylamide was grafted onto the backbone of cellulose extracted from waste newspapers by conducting microwave-assisted synthesis of polyacrylamide grafted cellulose (PAM-*g*-cellulose). The cellulose was extracted from the waste newspaper by a chemical treatment involving alkaline and bleaching treatment. The characterization result by Fourier-transform infrared (FTIR) spectrophotometer. The grafted copolymers have been synthesized by varying the ratio of cellulose to acrylamide. Scanning electron microscopy and FTIR confirmed the successfulness of copolymer grafting. The flocculation studies of the PAM-*g*-cellulose were evaluated in a synthetic kaolin suspension using the standard jar test method at various flocculant dosages, initial kaolin concentration, and pH conditions. It was found that PAM-*g*-cellulose with a molar ratio of cellulose to acrylamide is 1:7 exhibited a highly effective flocculation capability (96.2%) as compared to other synthesized grafted copolymer. The results indicated that the flocculant dosage and pH affected the flocculation efficiency significantly. The optimum condition for the turbidity reduction of kaolin suspension was achieved at a flocculant dosage of 200 ppm in an acidic pH 3 with a decrease in turbidity levels of 82.8%. Overall, the results demonstrated the potential of PAM-*g*-cellulose extracted from waste newspaper to serve as eco-friendly flocculants in water treatment.

Keywords: Cellulose; Graft copolymer; Flocculation performance; Turbidity removal

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1. Introduction

Water pollution has become the most serious environmental issue in the world besides air pollution. Water pollution consists of water pollutants that can harm human health physically or mentally. Different types of water sources such as groundwater or surface water contain different water pollutants [1]. Water pollutants can be categorized into two groups: organic compounds such as fuels, waste from plants, and volatile organic compounds. Another

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group is inorganic compounds such as ammonia, chemical waste from factories, and discarded cosmetics [2,3].

Water pollutants can be discarded and controlled by different technologies in wastewater treatment industries to obtain pure water. Several technologies have been used widely in wastewater treatment, such as ion exchange, membrane filtration, precipitation, flotation, solvent extraction, adsorption, biological and electrolytic methods, coagulation and flocculation process ultrafiltration, softening, and sedimentation. The coagulation and flocculation process is commonly used in industry because it is low cost and simple to remove various contaminants from the water sources [4]. This process gives high efficiency of removal suspended and dissolved solids wastewater treatment compared to others [5]. The coagulation and flocculation process is commonly used in wastewater industries such as palm oil mill effluent, textile wastewater, pulp mill wastewater, oily wastewater, and sanitary landfill leachates.

Coagulation and flocculation methods in industries use flocculants as the main chemical to trigger the agglomerate process of the small particles into large floc that needs to settle down [6]. There are two categories of flocculants that are available and conventionally used in wastewater industries: polymeric and inorganic flocculants. Natural and synthetic polymers are the components underneath the group for the polymeric group. Polyacrylamide is one of the common polymeric flocculants which easily accessible for synthesis [7]. In contrast, synthetic polymer possessed some disadvantages due to its sheer degradability and non-biodegradability. Natural organic flocculants based on natural polymers and polysaccharides are given attention since they are natural products and possess an environmentally friendly behaviour [5]. Natural polymers are the opposite of synthetic flocculants as not only are they fairly shear-stable, but they are also biodegradable and are easily available [8]. However, the advantage of natural polymer biodegradability becomes a drawback in reducing its storage life. Inorganic flocculants such as aluminium sulfate (alum), aluminium chloride, ferric sulfate, ferric chloride, poly aluminium chloride (PACl), aluminium chlorohydrate, and magnesium sulfate are the substances that have been used for decades in the flocculation–coagulation process. Previous studies state that using these inorganic flocculants gives numerous volume of metal hydroxide sludge, increasing the disposal and concentration of metal ions in treated water, affecting human well-being lifestyle [5,9].

Although natural and synthetic polymers had their pros and cons, graft copolymers have been introduced. Graft copolymerization is the modification of natural polymer that does not change its natural properties but improves in natural polysaccharide backbone rigidity of its composition after the purification process. As recent studies found a better way to produce eco-friendly flocculants, the combination of synthetic and natural polymers using the grafting method has improved the functional properties of flocculants synthesized from cellulose. The grafting of cellulose can be more efficient as it consists of a combination of synthetic composition polymer and natural polymer; it is more biodegradable and less toxic [10], without affecting the basic properties of cellulose. The microwave irradiation method is preferable for the grafting method because the microwave is much easier to obtain than using the commercial-free radical grafting method. The microwave-assisted method is used due to the highest grafting percentage efficiency because of its synergism in grafting by ring-opening under the free radical initiation [11].

The waste newspaper is one of abundance natural cellulose material. It consists huge amount of cellulose, lignin, and hemicellulose [12]. Besides using waste newspaper as an alternative natural flocculant, it can also reduce the accumulation of newspaper generated annually [13]. Waste newspaper is used as raw material in the synthesis of grafted polymer because it is easy to find anywhere. It is a low-cost material, besides it can reduce the insufficient landfill problem of the generated waste newspaper each year. The development of graft copolymer flocculants by utilizing other materials has been widely carried out. However, the use of old newspapers as flocculants in wastewater treatment is still minimal, so it is very feasible to continue developing. Thus, this study aims to develop graft copolymer flocculants by using waste newspapers as the source of cellulose and polyacrylamide by using the microwave-assisted method.

2. Materials and methods

The waste newspaper was collected at a residential area nearby Kampus Uniciti Alam, Sungai Chuchuh, Kangar, Perlis, Malaysia. Sodium hydroxide (NaOH), sodium chlorite ($NaClO_2$), sulphuric acid (H_2SO_4), ceric ammonium nitrate (CAN), kaolin powder, acetone were purchased from Sigma-Aldrich (USA). AWS/4D Aquamatic Water Stills Hamilton (UK) prepared double distilled water used throughout the experiment.

2.1. Extraction of cellulose from waste newspaper

In this study, the cellulose was extracted from waste newspaper by bleaching and alkaline treatment. Firstly, the waste newspapers were cut into small pieces and dried under the sunlight. The dried shreds of waste newspaper then were immersed in distilled water for 3 d. The water was changed frequently during this process. After that, the waste newspapers were boiled for around 15 min, and then it was washed several times by tap water. Next, the waste newspapers were undergone 4% of NaOH solution at 70°C for 2 h. The samples were treated using 1.7% w/v acidified sodium chlorite ($NaClO_2$) about 75°C for 4 h at pH 4.5. For the alkali treatment, the step was repeated 3 times, while for the bleaching treatment, it was repeated twice. For both treatment steps, the ratio of waste newspaper to liquor is 5:200 (g/mL). The sample was washed several times using distilled water to retain neutral pH [14]. The samples were dried using a hot air oven at a temperature of 65°C until a constant weight was obtained.

2.2. Synthesis of polyacrylamide grafted cellulose

An amount of the extracted cellulose dissolved into 4 mL of distilled water. The desired amount of polyacrylamide was dissolved in 1 mL and was added to the extracted cellulose solution. The mixed well of the mixture was then transferred to the crucible. Then, the CAN initiator is mixed together with the mixture solution. After that, the crucible was placed on a turntable microwave oven with microwave irradiation of about 800 W of power was performed. The microwave irradiation was paused before the mixture inside the reaction vessel started to boil (around 65°C), then the crucible was put in the ice water. This step is essential because it reduces the possibility of the mixture being splashed out from the crucible and prevents the formation of the vapors that contain acrylamide that is poisonous [15]. The microwave irradiation-cooling process was repeated until a gel-like mass was left. When the microwave irradiation process was completed, the reaction vessel was left to cool down for 24 h undisturbed to complete the grafting process. After the grafting reaction was completed, the sample was poured into an excess of acetone (25 mL). The resulting grafted copolymer was put into the hot oven to be dried at a temperature of 65°C for 4–5 h. The precipitate was pulverized and sieved. The effect of the molar ratio of cellulose to polyacrylamide was studied (1:1, 1:.7, 7:1). The CAN as initiator dosage was fixed at 0.05 g and reaction time fixed at 3 min. However, the molar ratio of cellulose to polyacrylamide was varied. The percentage grafting was calculated as follows:

$$\text{Grafting}\left(\%G\right)=\frac{\begin{array}{c}\text{Weight of graft copolymer}\\ -\text{ Weight of cellulose}\end{array}}{\text{Weight of cellulose}} \tag{1}$$

2.3. Jar test experiment

The experiment was carried out using a 1 L beaker with six paddle stirrers. 50 mg of kaolin powder was freshly prepared into 500 mL distilled water before the experiment started. Jar test was conducted with the initial period of rapid mixing at 200 rpm for 3 min, followed by slow mixing at 50 rpm for 30 min then sedimentation process about 30 min [16]. The supernatant was collected and analyzed its turbidity reading by using Turbidimeter (Model: HACH 2100N). Several parameters such as the molar ratio of cellulose to acrylamide, flocculants dosage, kaolin concentration, and pH of the kaolin suspension were studied in the flocculation process. 1 M of H_2SO_4 and 1 M HCl solutions were used to adjust the pH of the suspension.

The result of these parameters was calculated by using Eq. (2):

$$\text{Turbidity removal}\left(\%T\right)=\frac{T_1-T_2}{T_1}\times 100 \tag{2}$$

where T_1 (NTU) and T_2 (NTU) are the turbidities of kaolin suspension before and after the coagulation–flocculation process, respectively.

2.4. Characterization procedure

The raw waste newspaper, extracted cellulose, and polyacrylamide grafted cellulose (PAM-*g*-cellulose) were characterized using Fourier-transform infrared spectroscopy (FTIR). The FTIR (Model: Perkin Elmer) was used to determine the functional group present in the extracted cellulose. The extracted cellulose was recorded in the solid-state using the KBr pellet method with an FTIR spectrophotometer in 4,000–400 cm^{-1}.

3. Results and discussion

3.1. Extraction and characterizations of cellulose

The cellulose was extracted from waste newspaper by the chemical treatment which consists of immersion in alkaline solution and bleaching treatment. After conducting this treatment, the total weight loss was 53%, which demonstrates that only half of the total content was cellulose. The observation of colour change of the waste newspaper before and after pre-treatment proves the pre-treatments effectiveness. The waste newspaper colour changes from dark grey to a clear grey color and has a more cotton-like structure. The colour changes were due to removing non-cellulosic components such as lignin, hemicellulose, ink, and wax. Fig. 1 shows the waste newspaper before and after the pre-treatment.

The FTIR spectra of raw waste and extracted cellulose newspapers are shown in Fig. 2a and b, respectively. Fig. 2a shows the raw waste newspaper had stretching vibration of O–H bond assigned by the peak at 3,749 cm^{-1}. The band at 2,855 cm^{-1} peak in the raw waste newspaper had shown the stretching vibration of the C–H bond. The C=O bond of hemicellulose compound could be seen at

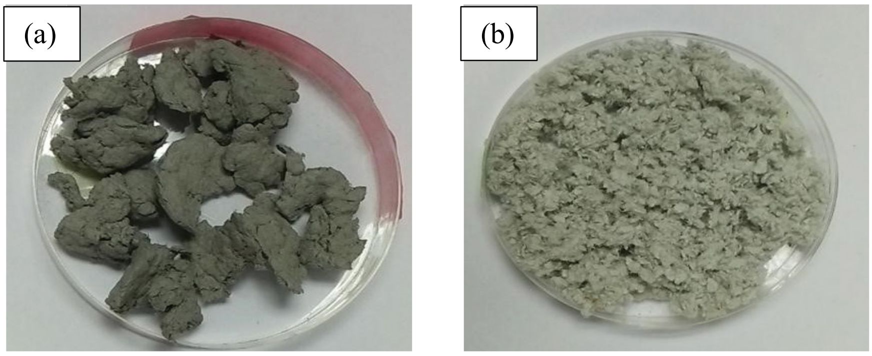

Fig. 1. Photo images of (a) waste newspaper and (b) extracted cellulose samples.

peak 1,735 cm^{-1}. Meanwhile, the lignin compound had present in the raw waste newspaper component can be seen at the peak of 1,505 cm^{-1}, which shows the C=C bond of the aromatic compound. The –C–O–C– a bond could be determined by peak 1,266 cm^{-1} showing the lignin's ether linkage. Previous studies [15,17] show the range peaks of the cellulosic bond similarly to this research. The lignin and hemicellulose peaks such as C=O, C=C, and –C–O–C– bonds disappeared in the extracted cellulose spectrum.

3.2. Synthesis and characterizations of PAM-grafted-cellulose by microwave-assisted method

The best grade of PAM-grafted-cellulose has been determined through its higher percentage of grafting. Based on Table 1, the best composition of graft copolymer as its highest percentage grafting is for the PAM-grafted-cellulose 1, which is the molar ratio of cellulose to acrylamide was 1:7. This graft copolymer occurs when the Ce IV from the ceric ammonium nitrate (CAN) generates its free radical to the backbone of the cellulose and forms a chelate complex with the hydroxyl group of cellulose and oxidant. The PAM aid the grafting process by elongating its bond as the microwave irradiation introduces and rotates its molecules into an elongation of the C–C double bond of the PAM. Then the pi bond splits up into two localized bonds, which offer the free radical sites on respective carbon. The free radicals combine with the cellulose backbone (aid with CAN) and the acrylamide molecule (aid with microwave radiation), which interact together through a common free radical step mechanism to produce the graft copolymer. The previous study by Kumar et al. [18] also provided a similar result on molar ratio cellulose to acrylamide. Hence, this data can be confirmed that this grafting method produces high yields of grafting products.

The FTIR spectra of extracted cellulose from waste newspaper and PAM-grafted-cellulose have been recorded by wavenumber in the range of 4,000–400 cm^{-1} using an FTIR spectrophotometer. Fig. 3a shows the FTIR spectrum of extracted cellulose of waste newspaper after chemical treatment; meanwhile, Fig. 3b shows the FTIR spectrum for PAM-grafted-cellulose after grafting with acrylamide.

As presented in Fig. 3, the peak of 3,426.32 cm^{-1} is attributed to the O–H stretching band of the hydroxyl group of cellulose in the waste newspaper. The additional peak of the CH_2 group for cellulose presence is clearly at peak 1,423.60 cm^{-1}. The –CONH– bonding between the PAM and the extracted cellulose has been done successfully, in which the C=O bond of cellulose and N–H stretching bond was present through the peak at 1,654.7 and 1,504.48 cm^{-1}, respectively [19]. Further, there is another peak at 1,383 cm^{-1} which is designated to the C–N stretching [15]. Comparing the two spectrums, it was confirmed that the grafting of polyacrylamide to the backbone of cellulose was successful.

The surface morphology of the grafted cellulose was studied by applying scanning electron microscopy (SEM). The SEM micrographs of the extracted and grafted

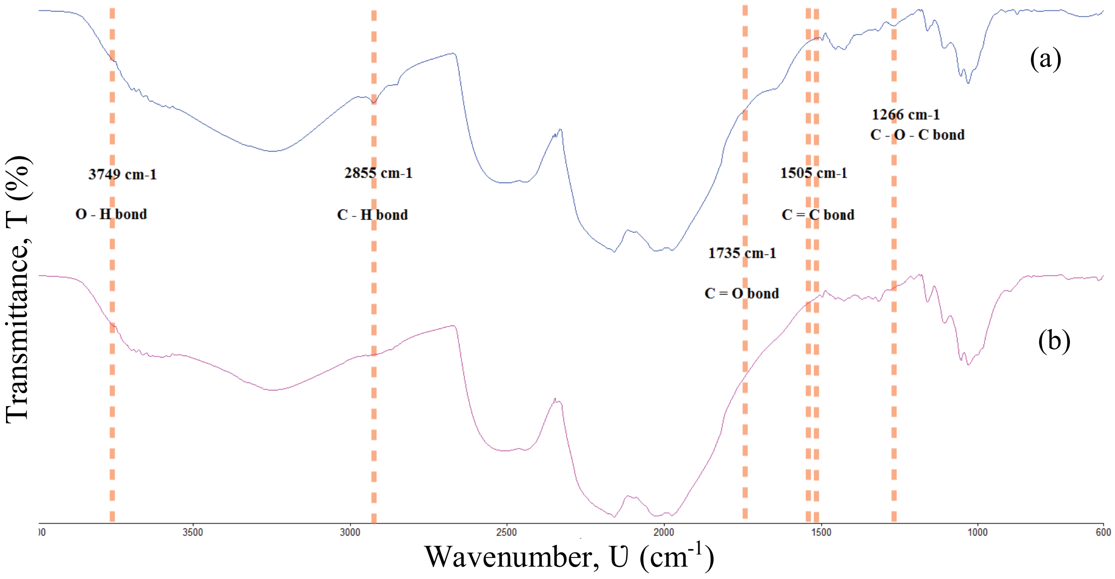

Fig. 2. FTIR spectra of (a) waste newspaper and (b) extracted cellulose.

Table 1
Synthesis details of PAM-grafted-cellulose

Grades	PAM-grafted-cellulose 1	PAM-grafted-cellulose 2	PAM-grafted-cellulose 3
Molar ratio cellulose to acrylamide	1:7	1:1	7:1
Percentage grafting (%G)	413 ± 0.55	40 ± 0.01	17 ± 0.14

cellulose are illustrated in Fig. 4. The morphological shape of the extracted cellulose from the waste newspaper was in a fibrillar structure with the glucose moieties chain. A change in the shape of the fibrillar structure of the extracted cellulose on grafting and thick polymeric coating of acrylamide on the surface of the PAM-grafted-cellulose shows that the opening chain of the extracted cellulose already closed by grafting with acrylamide [18]. After grafting, the morphological shape was changed into a granular lace structure. It shows that the PAM-grafted-cellulose was already grafted with polyacrylamide, which transformed into lacy morphology.

3.3. Flocculation performance of PAM-grafted-cellulose with kaolin suspension

3.3.1. Effect of different types of flocculants

In this research, the performances of the synthesized PAM-grafted-cellulose were tested on kaolin suspension using a standard Jar Test. The flocculation performances of the synthesized flocculants were evaluated at a different molar ratio of cellulose to acrylamide with a 200 ppm flocculant dosage. In addition, the non-grafted cellulose and polyacrylamide were also tested in this process with a similar condition.

As shown in Fig. 5, PAM-grafted-cellulose 1 shows the highest turbidity removal for the flocculation test, which achieved the maximum performance at 82.8%. Another synthesized PAM-grafted-cellulose with a different molar ratio also gives a higher percentage on turbidity removal. PAM-grafted-cellulose 2 and PAM-grafted-cellulose 3 also succeeds to remove turbidities at the percentage of 76% and 68.9%, respectively. These performances show that the higher the percentage of grafting of the synthesized PAM-grafted-cellulose, the higher the percentage of turbidity removal of flocculation on kaolin suspension. For the acrylamide flocculant, the percentage turbidity removal was 29.8% and for non-grafted cellulose itself, it could remove

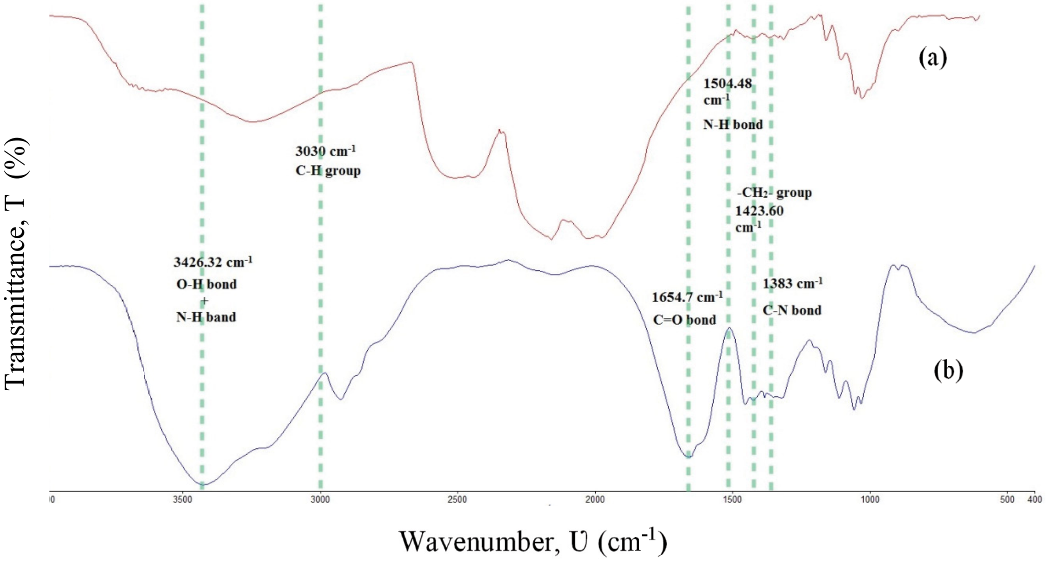

Fig. 3. FTIR spectra for (a) extracted cellulose from waste newspaper and (b) PAM-grafted-cellulose.

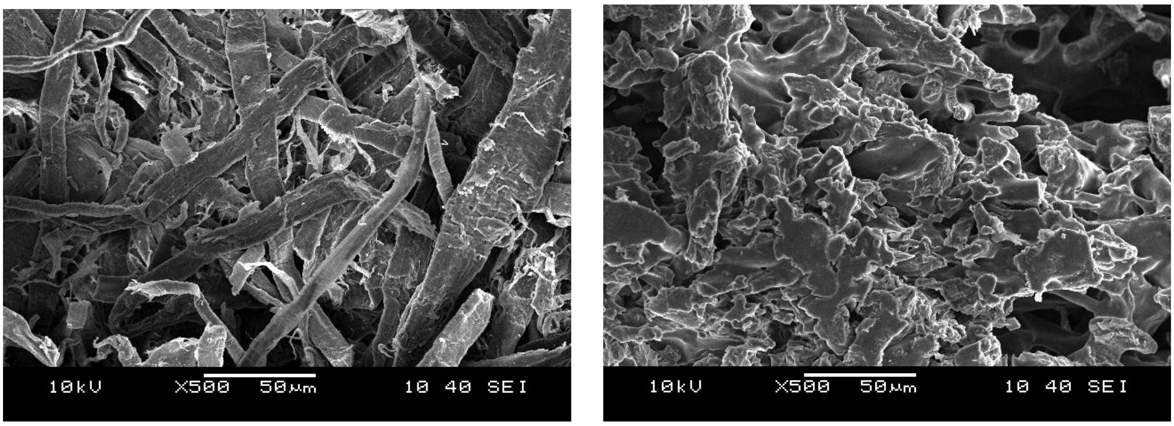

Fig. 4. SEM images of extracted cellulose (a) and PAM-grafted-cellulose (b) samples.

about 18.4% turbidity removal for the flocculation of kaolin suspension. This evidence shows that synthesized PAM-grafted-cellulose 1 had better turbidity removal than commercial flocculant polyacrylamide in the flocculation process.

3.3.2. Effect of flocculant dosage

PAM-grafted-cellulose 1 was chosen as it gives the highest turbidity removal compared to the others. The concentration of kaolin suspension was fixed at 200 ppm and the pH of kaolin suspension was constant at its original pH (~6–7). The range of flocculant dosage of PAM-grafted-cellulose 1 was set about 20 until 250 ppm.

Fig. 6 presents the trend of the graph on different amounts of flocculant dosages of PAM-grafted-cellulose 1. It was increased until it reached 200 ppm dosage and decreases when further increased flocculant dosage up to 250. PAM-grafted-cellulose 1 shows the maximum performance on removing turbidities (82.8%) at flocculant dosage 200 ppm. The optical density of supernatant collected at this dosage was decreased. This small amount of PAM-grafted-cellulose could lead small particles into large floc, which is ready to be settling down in the flocculation phase.

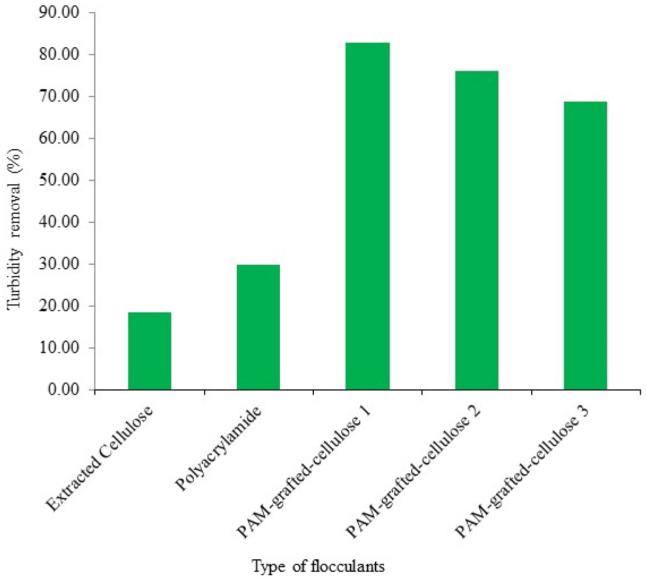

Fig. 5. Effect of different flocculants on percentage turbidity removal.

As the flocculant dosage further increased to 250 ppm, it still gives a high percentage of turbidity removal (80.6%), however, the PAM-grafted-cellulose 1 shows its decreasing trend. This is due to the overdosing of the flocculant, where the excess polymer is adsorbed on the colloidal surface and produces destabilized colloids [15,19]. Thus, the best dosage of PAM-grafted-cellulose 1 could initiate small particles to be removed at 200 ppm.

3.3.3. Effect of pH

The effect of pH on kaolin suspension was an important part of this research. It measures which condition of synthesized PAM-grafted-cellulose 1 could better remove suspended solids. The results are presented in Fig. 7.

The best pH of kaolin suspension occurred at pH 3, which is at the acidic condition with the highest percentage turbidity removal of 96.2%. In acidic conditions, much bigger flocs were formed due to the bridging effect made by the particles surrounding the surface of PAM-grafted-cellulose. Besides, the collected supernatants' final result could be shown in Fig. 8. The treated supernatant at pH 3 became cleaner and more transparent than another kaolin suspension pH.

The turbidity declined significantly when the pH was increased until pH 11. At pH 11, the turbidity reached almost zero percent on turbidity removal because PAM-grafted-cellulose's performance was not efficient in higher pH of kaolin suspension. This happened because the kaolin particles are similar charge ions with the PAM-grafted-cellulose, so they repelled to each other and did not form any flocs. The cloudiness of the final flocculation at pH 11 did not change after putting 200 ppm of PAM-grafted-cellulose 1, which can be seen in Fig. 8.

3.3.4. Effect of kaolin concentration

In this study, the flocculant dosage of PAM-grafted-cellulose 1 was set at 200 ppm and the condition of pH 3

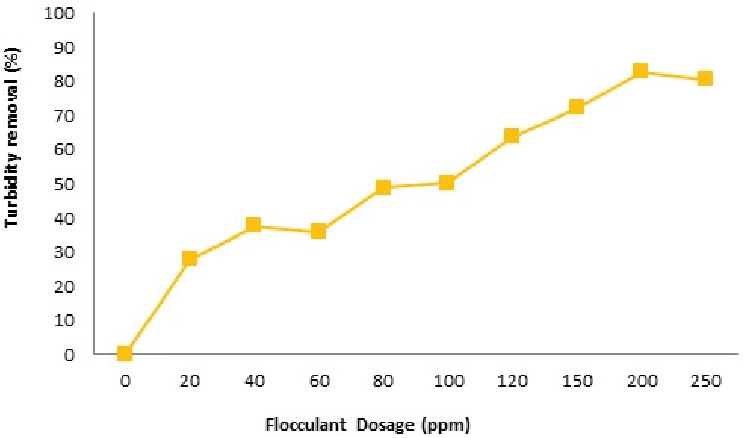

Fig. 6. Effect of flocculant dosage on percentage turbidity removal.

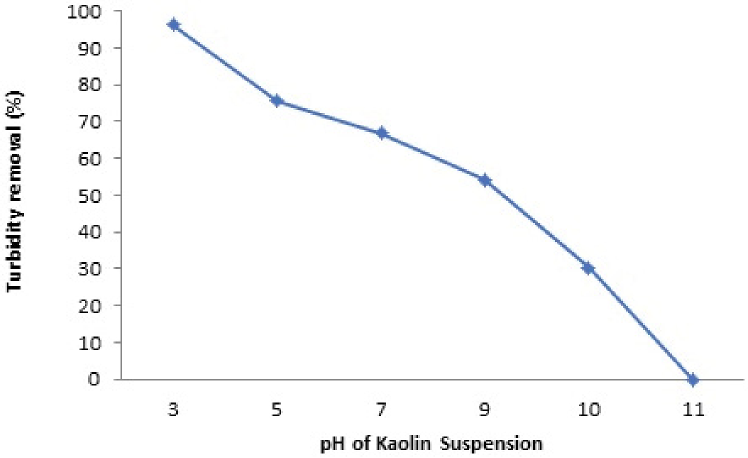

Fig. 7. Effect of pH on percentage turbidity removal.

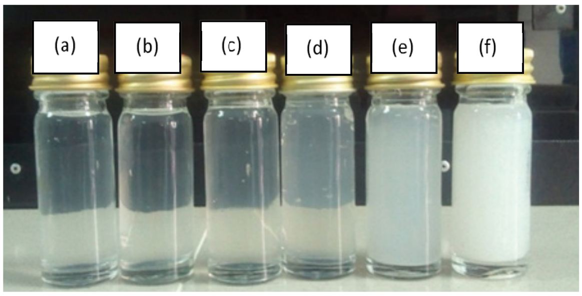

Fig. 8. Supernatants of different pH of kaolin suspension at (a) pH 3, (b) pH 5, (c) pH 7, (d) pH 9, (e) pH 10 and (f) pH 11 after flocculation process.

was fixed throughout this experiment. The initial concentration of kaolin suspension was varied from 50 to 2,000 ppm. The results are presented in Fig. 9. The results show that the flocculation performance of the PAM-grafted-cellulose 1 was intensely increased as the initial kaolin concentration reached 200, which resulted in 88.1% turbidity removal. However, as the initial kaolin concentration further increased up to 2,000 ppm, the performance of the PAM-grafted-cellulose was slightly decreased. These results proved that the flocculants dosage needs to be controlled in a suitable range of initial kaolin concentration to achieve better performance [20].

4. Conclusions

The cellulose was extracted from waste newspaper by using chemical treatment. The FTIR spectra proved that the cellulose was successfully extracted from the waste newspaper. Polyacrylamide grafted cellulose (PAM-grafted-cellulose) was synthesized by microwave-assisted method. The PAM-grafted-cellulose 1 with the highest percentage grafting (413 ± 0.55) gives the maximum percentage of turbidity removal in flocculation of kaolin suspension. The best grade of PAM-grafted-cellulose showed the highest percentage turbidity removal at pH 3 and the best flocculants dosage was 200 pm.

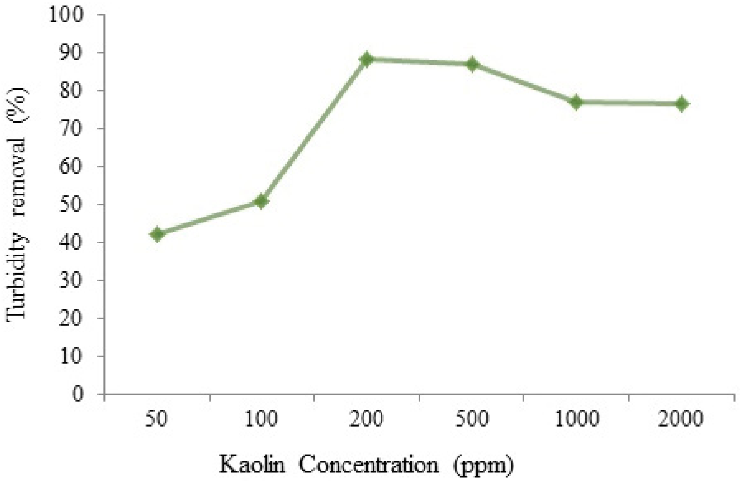

Fig. 9. Effect of kaolin concentration on percentage turbidity removal.

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Conflicts of interest

The authors declare no conflict of interest.

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