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Jackfruit Seeds Starch-Based Coagulant for Synthetic Textile Wastewater Remediation

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Abstract: The chemical-based coagulants are commonly used in wastewater treatment due to its effectiveness. However, their lack of biodegradability and health hazard of the monomer residues are the main concern. Thus, starches isolated from fruit wastes and plants which rich in polysaccharides were investigated as a potential alternative as natural coagulant. In this study, jackfruit seed starch (JSS) coagulant was produced by isolating starch from jackfruit seeds using alkaline pretreatment. Physicochemical properties of JSS were studied including starch yield, amylose to amylopectin ratio, functional group, surface morphology and compositional elements. In order to assess the effectiveness of JSS coagulant, a coagulation-flocculation process was performed to observe colour and turbidity removal from synthetic textile wastewater. The parameters of coagulation-flocculation conducted are pH and coagulant dosage. It was found that the optimum parameter for coagulation-flocculation process are at pH 2 and coagulant dosage of 50 mg/L. At pH 2, 95.5% of colour and 87.5% of turbidity were removed. In addition, colour removal was up to ~100% and turbidity removal was 89.1% when 50 mg/L JSS coagulant was used. The JSS flocs were filtered and analyzed to identify chemical functional group, surface morphology and compositional elements by FTIR, SEM and EDX analysis. The most abundant functional group in JSS coagulant which is –OH group, played important roles in this coagulation-flocculation process. Thus, this jackfruit seed coagulant can be one of the options of natural coagulant to be used in wastewater treatment

Keywords: Jackfruit Seed Starch, Natural Coagulant, Textile Wastewater, Colour, Turbidity

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1. Introduction

Textile industry has become one of the world's worst delinquents in terms of toxic waste because it requires a great amount of two components which are water and chemical. The chemicals used are includes strong acids, strong alkalis, inorganic chlorinated compounds, sodium hypochlorite. Other than that, organic compound such as dyestuff, bleaching agent, finishing chemicals, starch, thickening agent, surface active chemicals, wetting and dispersing agents and salts of metals are also in the list [1]. Therefore, the wastewater discharged are commonly polluted with high concentration of by-products from the process. The high colouring power of dyes can cause aesthetic damage on the potable water [2].Coagulation-flocculation is a common wastewater treatment as it is one of the simplest and most efficient method. Coagulants are varies from chemical to non-chemical and could be from synthetic or natural sources. As reported in previous study, aluminium sulphate (alum) is most commonly used as it is easy to handle and cheap [3]. However, the coagulation by alum also have disadvantages which are high quantities of chemicals needed, produce high volume of sludge [4], and there is significant change in pH of treated water [5]. Nevertheless, uncontrolled coagulant administering, inefficient use of the coagulant, or issues in the chemical reactions of treatment process will cause the presence of residual monomers [6]. They are undesirable because of their neurotoxicity and strong carcinogenic properties [3].

In order to find the alternative for the use of chemical-based coagulant, many researches developed interest into natural coagulant which can be produced or extracted from plant, animal and also microorganisms. By considering the cost, safety for ecology system, human health, biodegradability, and a comprehensive acceptable operational effective dose range of flocculation for various colloidal suspensions, natural coagulants are most suitable to be used for wastewater treatment. Since the plants can be locally grown, they are also more cost-effective than imported chemicals [7]. Conventional starches and starches isolated from fruit wastes and plants which rich in polysaccharides are gaining increasing interests as potential natural coagulant. From previous studies, starches isolated from different sources are well documented for various applications. On the other hand, there are also many of their applications in food industries such as thickener and stabilizer [8] and also as tablet binders [9]. Besides, starches were also discovered by research for wastewater treatment as coagulant to remove turbidity [10, 11] and adsorbent for the removal of Rhodamine B dye in dye wastewater [16]. Starch can be used as natural coagulant due to its low cationic polyelectrolyte properties [12]. Therefore, in this study, jackfruit seeds starch (JSS) was isolated and characterized with respect to the physicochemical properties. Then, the efficiency of jackfruit seeds starches as natural coagulant to treat synthetic textile wastewater was evaluated.

2. Materials and Methods

2.1 Preparation and isolation of jackfruit seeds starch.

The jackfruit seeds were collected from fruit stall in Pekan Pagoh. The undesirable parts of the seeds such as the sprouting and the outer seed coats were also removed. The seeds were washed and rinsed. After that, the seeds undergo drying process in a pre-heated hot air oven to 65˚C for approximately 48 hours. The weight were measured triplicate at every hour to ensure constant weight is obtained [11]. The dried seeds were ground by using grinder and sieved by sieve shaker into the size ranging from 63 µm. The powders will be packed accordingly into zip-lock bags.The isolation of jackfruit seed starch was carried out according to the methods reported by [11].

The powdered seeds with weight of 1 part solid to 10 parts of 0.05 M NaOH was mixed at 200 rpm stirring speed. Then, the mixture was filtered using a four-fold muslin cloth to remove seed fibers and solid residues into an empty beaker. The removed solid residues undergoes alkaline extraction for the second times to isolate the remaining starch components. The filtrate was centrifuged at 4000 rpm, 25 °C for 10 min. The isolated starch formed a whitish paste after the supernatant drained and the brown sediment scrapped off. 0.1 M HCl neutralized the isolated starch to pH 7.0 ± 0.1. The remaining whitish sediment was washed with distilled water and left in the oven for overnight drying at 40 °C for complete drying. The dried powder was collected, pounded, packed in a zip-lock bag and stored in a desiccator until further analysis.

2.2 Preparation of synthetic textile wastewater

The synthetic textile wastewater was prepared according to method by [13]. 200 mg of dye powder (Congo red, L173) was dissolved into 1 L of distilled water making the solution into 200 ppm concentration. The dye with pH of 3-5 was supplied by Comak Chemical Product. The initial measurement of pH, colour and turbidity were measured and recorded.

2.3 Physicochemical analysis of isolated jackfruit seeds starch.

2.3.1 Determination of starch yield.

The percentage yield of isolated JSS was obtained by calculating using Eq. (1):

$$\text{Yield} = \frac{\text{Dry weight of isolated starch}}{\text{Dry weight of seed powder used for extraction}}\ x\ 100\% \qquad Eq.(1)$$

2.3.2 Determination of amylose and amylopectin content

The amylose and amylopectin content was determined by using a simple and rapid calorimetric method which adopted from [14]. 0.1 g of isolated JSS samples was dissolved by heating at 95°C for 10 minutes in mixture of 1 mL of 99% ethanol and 9 mL of 1M of NaOH. After dissolve, this solution was cooled before being diluted with distilled water in 100 mL volumetric flask. 5 mL of this solution was mixed with 1 mL of 1M acetic acid and 2 mL of iodine solution. The distilled water was added to the mark of 100 mL volumetric flask. The absorbance at 620 nm were measured using T60 UV-Vis Spectrophotometer (PG Instruments, UK). The amylose and amylopectin content were calculated using Eq. (2) and Eq. (3) respectively:

$$\text{Amylose content (\%)} = 3.06\ x\ \text{absorbance}\ x\ 20 \qquad Eq.(2)$$

$$\text{Amylopectin (\%)} = 100\ \%\ \text{of amylose content} \qquad Eq.(3)$$

2.3.3 Determination of chemical functional group

The presence of functional groups in the isolated JSS samples was determined using Spectrum Two FT-IR Spectrometer (Perkin Elmer, USA). The wavelengths was in range of 700 to 3700 cm^{-1}. The FTIR spectrum was expressed in terms of % transmittance.

2.3.4 Determination of surface morphology

The surface morphology of isolated JSS was analyzed using scanning electron microscope (SEM) EM-30AX (COXEM, Korea). This SEM verified the shape of the starch granules. The structure was observed at magnifications of 1000x and 2000x according to previous studies by [8]. The energy-dispersive X-ray (EDX) which equipped with SEM was used to analyze the information on the element present in the sample for the ease of quantification and chemical identification [11].

2.4 Coagulation and flocculation

The coagulation-flocculation studies was carried out using a conventional 4 jar apparatus or flocculator (VELP Scientifica JLT4 Flocculator, Italy) which adopted from [15]. The beaker filled with 250 mL of synthetic textile wastewater which adjusted to desired pH and added with different dosage of the isolated JSS coagulant. The mixture was mixed at 100 rpm 3 minutes (rapid mixing) and will be followed by slow mixing at 40 rpm for 15 minutes. After the agitation being stopped, the beakers were removed carefully and transferred to the flat surface to allow sedimentation for 30 minutes. The supernatant was filtered through a Whatman® filter paper (pore 45 µm) using vacuum filter flask. The treated and untreated synthetic textile wastewater were measured for their colour and turbidity to evaluate the effectiveness of the JSS coagulant. Calibrated turbidimeter (HACH TL2300, USA) was used to measure turbidity of the synthetic textile wastewater. The colour concentration was measured by using T60 UV-Vis Spectrophotometer (PG Instruments, UK) at 498 nm wavelength.

3. Results and Discussion

3.1 Physicochemical analysis

3.1.1 Starch yield and amylose to amylopectin ratio

The starch was isolated from jackfruit seeds by using alkaline extraction. The yield of the isolated JSS was calculated using Equation 3.1 was 19.42%. Meanwhile, the ratio of the two glucose polymers in starch which are amylose and amylopectin were determined by using a rapid calorimetric method. The distribution of these polymers are the utmost important for starch functionality as a natural coagulant [11]. The amount of amylose and amylopectin in this isolated JSS are 27.9% and 72.1% respectively.

3.1.2 Chemical functional group of JSS coagulant and floc

The chemical functional group of JSS coagulant, congo red (CR) dye and JSS floc were identified by using FTIR. Figure 1 shows the spectra obtained which indicates several main peaks at the wavelengths between 3700 cm^{-1} and 700 cm^{-1}. The spectrum of the JSS coagulant shows the major absorption peaks that correspond to different functional groups vibration. The JSS coagulant significantly broad peak at 3272 cm^{-1} which indicates the O-H stretching and distinctive peak at 1635 cm^{-1} which indicates O-H bending. The presence of these intermolecular hydrogen bonded hydroxyl groups in single bridge compound is due to contributions of water absorption in noncrystalline region of the starch [16]. Similar peak was obtained for JSS floc which is at 3298 cm^{-1}. However the percentage of transmittance was higher which means that the fewer bonds were available to absorb the IR because the CR dye were already bonded with the JSS coagulant. In addition, the peaks presented in range of 1000 to 1300 cm^{-1} for these three spectra were indicates the C-H bending [17]. Meanwhile, the peaks observed at 2930 cm^{-1} in JSS coagulant and 2867 and 2936 cm^{-1} in JSS floc were attributed to C-H stretching of aliphatic structures assigned to fatty acids and lipids [18]. The bending and deformation bands related to the carbon and hydrogen atoms were discovered in the region 1500-1300 cm^{-1} [19] as observed by the vibrational signal occurs in the range. Notably the peak at 1336 cm^{-1} was assigned to C-O-H bending. The peak presented at 2930 cm^{-1} in JSS coagulant was also attributes to the presence of amylose and amylopectin. The presence and stretching of C-O group with hydrogen bond were observed at the peak of 1150 and 1077 cm^{-1}.The presence of glycosidic bond (C-O-C) in amylose and amylopectin in JSS coagulant was observed at 928 cm^{-1}.

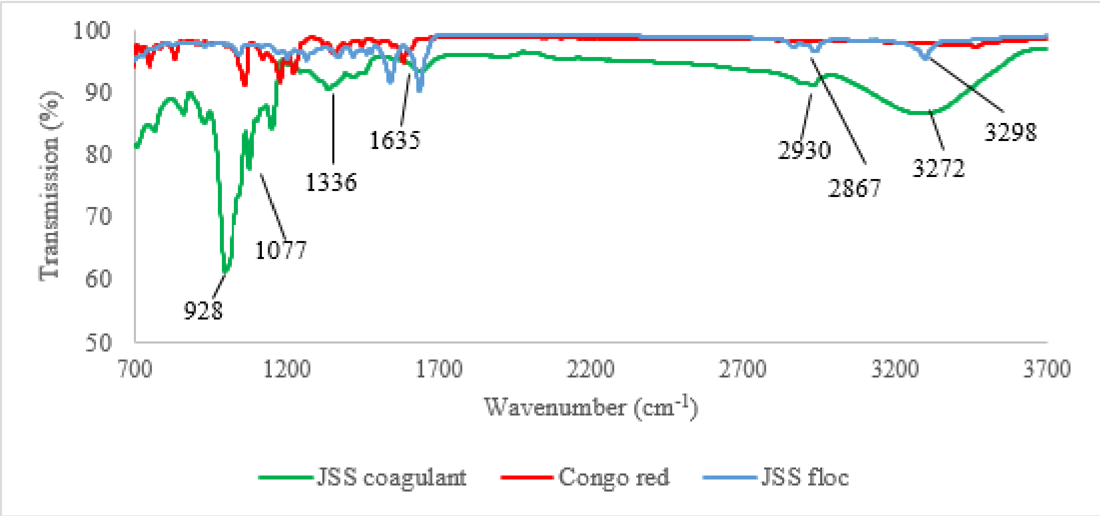
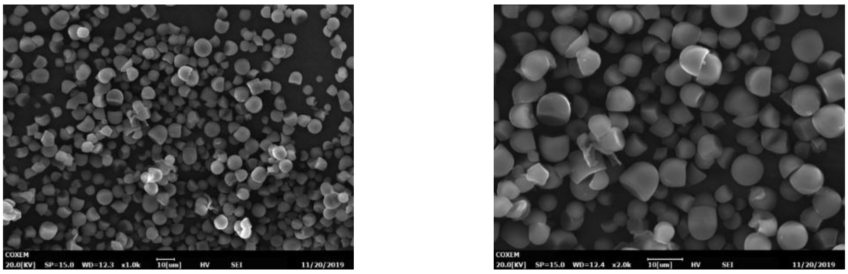


Figure 1: FTIR spectra of JSS coagulant, CR dye and JSS floc

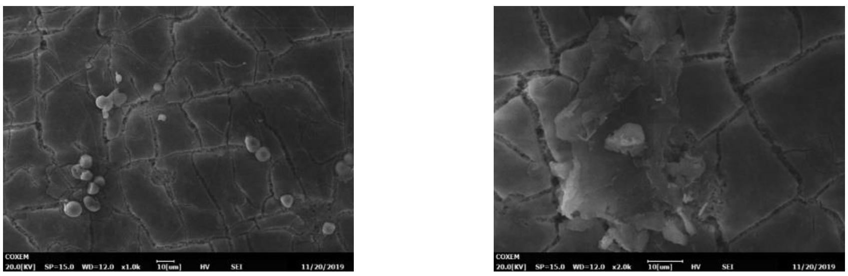
3.1.3 Surface morphological properties of JSS coagulant and floc.

The surface morphology of the JSS coagulant and floc? were obtained by using SEM. It was found that the JSS coagulant was in bell-shaped as shown in Figure 2 (a) and (b) which were observed at magnification 1000x and 2000x respectively. The surface of the starch granules was also observed to be smooth. The same result was obtained by [8, 20] which stated that the isolated starch granules were round to bell-shaped with smooth surface. The same magnification also used to identify the surface morphology of the JSS floc as shown in Figure 3 (a) and (b). The images show the surface of JSS floc which are rough surface and ruptured shape. These indicate that there are interactions occurred at the surface of the coagulant between the CR dye particles and surface of the JSS coagulant.



(a) (b)

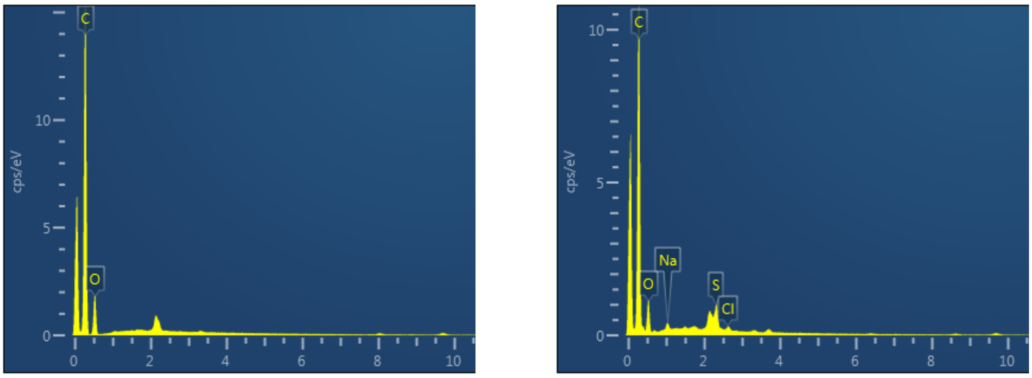
Figure 2: SEM image of JSS coagulant at magnification (a) 1000x (b) 2000x



(a) (b)

Figure 3: SEM image of JSS floc at magnification (a) 1000x (b) 2000x

The EDX analysis was performed to identify the composition elements in JSS coagulant and JSS floc. The main elements in JSS coagulant are carbon (C) and oxygen (O) as presented in Figure 4 (a).



(a) (b)

Figure 4: EDX analysis of (a) JSS coagulant and (b) JSS floc

Basically, starch is made up of a large number of glucose ($C_6H_{10}O_5$) units. However, EDX analysis did not detect the presence of H in starch granules because there is no core electrons in H but only valence electrons. [21] stated that the identification is not possible because the signals emitted from H 1s valence electrons would overlap with signals of other valence electrons. Meanwhile, the elements in JSS floc are consist of C, O, sodium (Na), sulfur (S) and traces of chloride (Cl). The EDX analysis for JSS floc was shown in Figure 4 (b). Among the various elements, C was found to be the most dominant in the JSS floc. The CR with chemical formula $C_{32}H_{22}N_6Na_2O_6S_2$ contain all the elements identified except for Cl which is from the hydrochloric acid (HCl) that being used to adjust the pH of the wastewater.

3.2 Coagulation-flocculation analysis

3.2.1 Effect of pH

In the coagulation process, the pH of solution has been identified as the most important parameter affecting dye as it affects the surface charge of the coagulant and also the stabilization of the suspension [22]. The Congo red (CR) dye is stable at pH range of 5 to 13. However, the pH is also known to affect the stability of the CR dye structure. At strong acidic condition, the red solution became dark blue due to the formation of protonated species that will neutralize the negative charge of CR dye. This will lead to destabilization and flocculation [23].

The coagulation of CR dye was studied with the initial concentration of synthetic dye wastewater was 200 ppm. Figure 5 (a) represents the percentage removal of colour and turbidity when treated with constant coagulant dosage which is 20 mg/L. From this graph, it is observed that the highest removal of CR dye was at acidic condition particularly at pH 2 with 95.5% of removal. At this pH, dye is exists as cation. The JSS coagulant is a polysaccharides that linked by glycosidic bonds and various side functions [23]. It contains hydroxyl groups (–OH) which are anionic and able to increase the coagulation competency. When charges are present, polysaccharides behave as polyelectrolytes. Cationic dye molecules get attached on the surface of coagulant thus neutralize and give particle attraction and hence coagulation occurred. As the pH increase, the percentage of colour removal reduced to 64.1, 56.3, 9.7 and 2.7% at pH 3, 4, 5 and 7 respectively. As for turbidity, the graph was plotted in Figure 4.6 to indicate the percentage of the removal. The percentage of turbidity removal were gradually decreased as the pH increased in the same trend as colour removal. At pH 2, the JSS coagulant managed to remove 87.5%. The percentage of removal reduced to 86.7, 69.7, 68.3 and 65.3% for pH 3, 4, 5 and 7 respectively.

3.2.2 Effect of coagulant dosage

Dosage of coagulant is one of the most important parameters that need to be consider to determine the performance of the coagulants in coagulation-flocculation for wastewater treatment. Figure 5 (b) illustrates the graph for colour and turbidity removal percentage of synthetic dye wastewater with 200 ppm initial concentration at pH 2. The results show that the increasing trend of colour removal as the coagulant dose increase. The isolated JSS coagulant with high amount of amylopectin provide abundant of ionic sites that will bind with anionic CR dye particles. Thus, the presence of a large amount of functional groups provides abundant adsorption sites that lead to the interparticle bridging [23]. Bridging of the polymers play a large part in the coagulation process as the higher the dosage of coagulant, the colliding particles will aggregate between themselves [24]. In this study, the increase of JSS coagulant dosage will increase the amount of amylopectin in the solution. These long-chain polymers will favor the bridging by giving the possibility of attachment of the ‘dangling’ polymer segments to other particles [25]. In this study, it was found that the percentage of the colour removal increase from 95.1% with 10 mg/L coagulant dosage to up to ~100% with increasing coagulant dosage. As discussed in previous section, the coagulation of CR dye is very effective at pH 2. Thus, to evaluate the effect of coagulant dosage, the pH was constant at pH 2. The results show that with 50 mg/L of coagulant dosage, the colour of the CR dye wastewater were able to be completely removed. The same increasing trend was obtained for turbidity removal (Figure 4.8) although the removal was not as high as for colour removal. The removal of turbidity was 85.4% with coagulant dosage at 10 mg/L. The removal increased to 88, 88.2, 88.4 and 89.1% for 20, 30, 40 and 50 mg/L respectively.

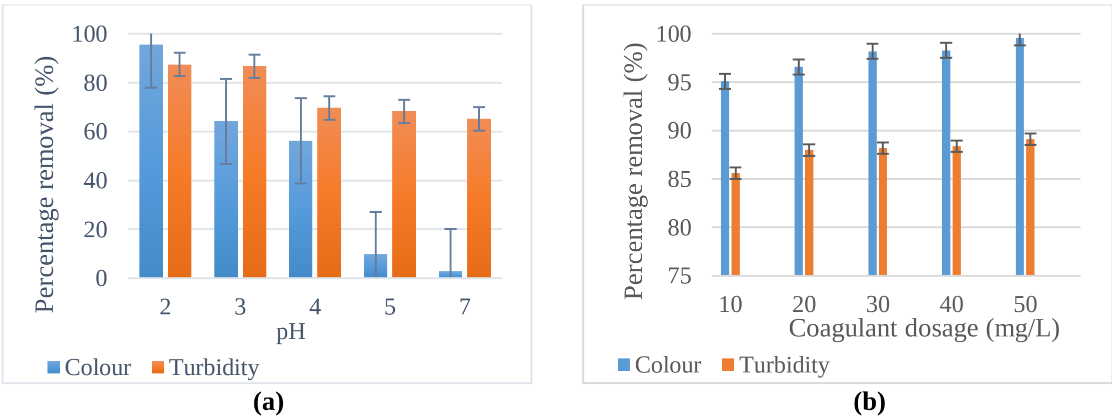


Figure 5: Percentage of colour and turbidity removal at parameter (a) pH (b) coagulant dosage

4. Conclusion

To conclude, the experiments conducted confirmed the positive coagulation properties of JSS as it contributed to colour and turbidity removal in synthetic textile wastewater. The removal of colour and turbidity were found to be the highest at pH 2 and coagulant dosage of 50 mg/L. The synthetic textile wastewater with initial concentration of 200 ppm was effectively removed up to ~100% colour and 89.1% of turbidity at the optimum condition. To confirm the coagulation occurred between the JSS coagulant and the CR dye, the characterization of the both were observed by FTIR, SEM and EDX analysis. The most abundant chemical functional group in JSS coagulant which is –OH group played important roles in this coagulation-flocculation process. Thus, this jackfruit seed coagulant can be one of the options of natural coagulant to be used in wastewater treatment

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