

# Literature Review: Fabrication Method, Characterization, Performance, and Application of Cassava Peel as Bio-Coagulant for Wastewater Treatment

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## Abstract

*The purpose of this study was to conduct a literature review to determine the understanding of current research on the use of Cassava Peel (CP) in the field of wastewater treatment, especially as a coagulant. This study reports on the types of coagulant modification of CP, coagulants characterization of CP for each modification, coagulants performance of CP under various conditions, and various applications of CP coagulant for wastewater treatment. This literature study takes references from 51 articles from the period 2011-2022. We mapped each article used and visualized it using the VOS viewer to find out the relationship between topics and absorption capacity in terms of article titles and keywords. From the results of a literature review study, CP is an attractive option and has the potential to be used as a natural coagulant or adsorbent to remove pollutants (such as dyes and turbidity) in wastewater treatment. The results of this study are expected to provide benefits and ideas for utilizing agricultural waste such as CP to become an economically efficient material.*

**Keywords:** Adsorbent, Agricultural waste, Cassava peel, Economical coagulant, VOS viewer, Water treatment.

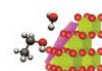
## 1 Introduction

Clean water is one of the basic needs that is very important for humans, especially in fulfilling domestic and industrial activities. The need for clean water increases with population growth every year. This phenomenon is in line with the unavailability of safe clean water. By 2050, more than half of the global population (57%) of people will live in a country or region that will experience a water crisis [1].

Environmental pollution, especially water pollution, is a problem in the availability of clean water. Examples of common environmental contaminants are heavy metals such as mercury, lead, copper, etc. [2]. Water pollution resulting from various human activities including domestic, industrial, and agricultural activities. If consumed in low doses, it will endanger human health and

the environment. Therefore, water treatment with the aim of removing contaminants in it is crucial to do as an effort to provide clean water for various needs.

Based on the method, the wastewater treatment process is divided into three types, namely physical, biological, and chemical treatment. Chemical water treatment methods, consisting of coagulation and flocculation [3]. Coagulation and flocculation technology into important components in conventional water treatment systems designed to remove impurities in water [4]. The types of coagulants and flocculants that are often used are polyaluminum chloride and synthetic polymers (polyacrylamide). However, excessive use of these substances can lead to various environmental consequences, as well as exhibiting neurotoxicity and carcinogenic



effects [5]. Therefore, the dose of chemical coagulant used needs to be controlled to reduce residual ingredients in the product which can then carry negative chemicals to water consumers.

The development of 'green' water treatment methods is a fairly interesting topic, because it involves biopolymer materials [4]. The use of environmentally friendly biocoagulants or biofloculants can be an alternative solution [6]. Biopolymer is an interesting concern because it is a natural product that is environmentally friendly with low production costs [7]. The development of biopolymers that are used as coagulants need to pay attention to the side of effectiveness, availability in nature in abundance, environmentally friendly, and economically feasible. Among the available biopolymers are chitin, chitosan, plant tannins, algae alginate, and plant starch. One of alternative coagulant that has high potential to remove organic content, turbidity, and total suspended solid is Cassava (*Manihot esculenta*) [4]. The most common applications of cassava are in starch extraction [8], sewage and wastewater treatment [9], dye adsorption [10], bioactive compounds [11], and ethanol biofuel production [12]. The high application of cassava peel as a coagulant aid is due to the molecular structure with functional groups having high affinity to agglomerate with other elements and impurities in water [13].

In addition, cassava starch granules consist of amylose (20%) and amylopectin (80%) [14]. Therefore, the sludge produced from the coagulation process by vegetable coagulants, including cassava, is cost-effective, highly biodegradable, non-toxic, non-corrosive and unlikely to produce air with extreme pH [15]. In addition, natural coagulants are relatively inexpensive to obtain and are available locally.

In the processing of cassava-based foods, cassava tubers are usually peeled to remove the outer layer which consists of the periderm, the cortex, which is the thin inner layer, and some thin cortex. Generally, cassava peels are discarded and left to rot [16]. Another fact is that the composition of cassava peel can reach 20-35% of the total weight of cassava tubers. The conversion of this by-product into an effective coagulant aid

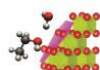
increases the market value of the research which will increase the market value and ultimately benefit the producers [17].

Cassava peel contains polysaccharides such as starch, pectin, and cellulose. It was seen that pectin, starch, and cellulose contain many carboxyl, hydroxyl, and amino groups which have significant potential for metal adsorption. In addition, functional groups with effective charge can help alum to form larger flocs in the coagulation and flocculation processes.

Inorganic salts, polymeric coagulants, organic polyelectrolytes, and composite inorganic-organic coagulants are among the coagulant materials used, and cost and storage are important considerations in addition to optimizing removal efficiency. Commonly, inorganic polymeric coagulants are used in water treatment due to their superior coagulation behavior and ease of preparation, whereas reagents with low molecular weight have a lower aggregation capacity. The inherent advantages of organic coagulants are less dosage, less sludge volume, and less pH dependence when compared to inorganic coagulants, but their high costs severely limit their widespread use in water treatment. Recently, research has been devoted to the preparation of inorganic-organic coagulants by combining the advantages of individuals in order to seek more effective, cheap, and stable coagulants.

This article shows the results of a literature review that explains the effectiveness of cassava peel as a coagulant in water treatment processes in general with a focus on characterization, optimization of several water treatment parameters, and applications in the treatment of several types of water. This literature study refers to 51 articles from the year 2011-2022.

Data analysis in this study is also equipped with a mapping tool to analyze the development of research related to cassava peel as a coagulant for wastewater treatment. Bibliometric analysis to produce network visualization of co-worker maps and co-worker density maps was performed using VOS viewer software [18]. Trend analysis from this research was conducted through a Google Scholar search with the keyword "Cassava Peel Coagulant" during the period 2017-2022. From 2017-2022, there were 382 articles related to cassava peel-based natural coagulant topics that were collected from the Google Scholar database. Then, Microsoft Excel 2010 was used to analyze data in the form of the number of publications per

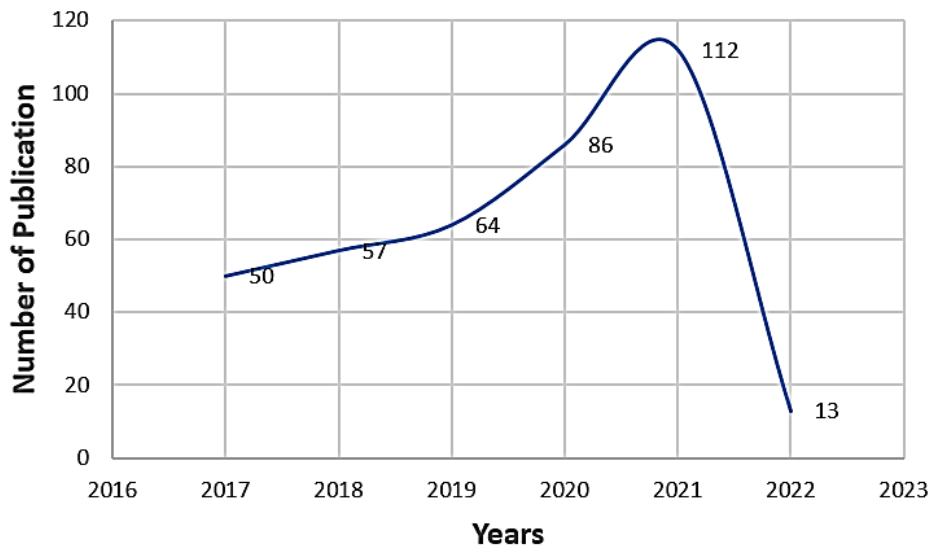


year. Meanwhile, the VosViewer software was used to analyze trends in the development of international publications on cassava peel area. **Figure 1** shows the development of research in the cassava peel area. **Figure 1** shows that the growth of studies on cassava peel is increasing every year. The most significant increase was in 2021 when the number of articles on the study of cassava peel reached more than 100 articles. However, in 2022 there was a significant decrease, in which the number of articles was no more than 15 articles. The decline in 2022 was due to data collection at the beginning of the year (January 2022).

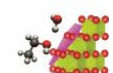
Then, **Figure 2** shows the results of the analysis based on the network visualization obtained from VOSviewer. VOSviewer is used for co-word mapping analysis based on setting the minimum number of occurrence of keywords is 4 and the threshold is 357. Based on Figure 2, the research area in the cassava peel area contains 9 clusters. The number of keywords found correlated with the larger the size of the circle shape in each cluster. Based on Figure 2, the

keyword "cassava peel" has the largest circle shape than other keywords which shows that the keyword "cassava peel" is the highest number compared to other keywords.

Another way to view a co-word map or keyword map on research developments in a specific field of science is through density visualization [15]. **Figure 3** depicts a high-level overview of keyword density in cassava peel research. The density of keywords or co-words is depicted using color in this visualization map. The greater the density of the colors depicted on the map, the greater the density of the keyword volume. The smaller the keyword density, the thinner the displayed color. According to the density visualization map in **Figure 3**, the most researched topic is "cassava peel," which is visualized with the most concentrated yellow color when compared to other topics.



**Figure 1.** The growth of international publications on cassava peel research topics.



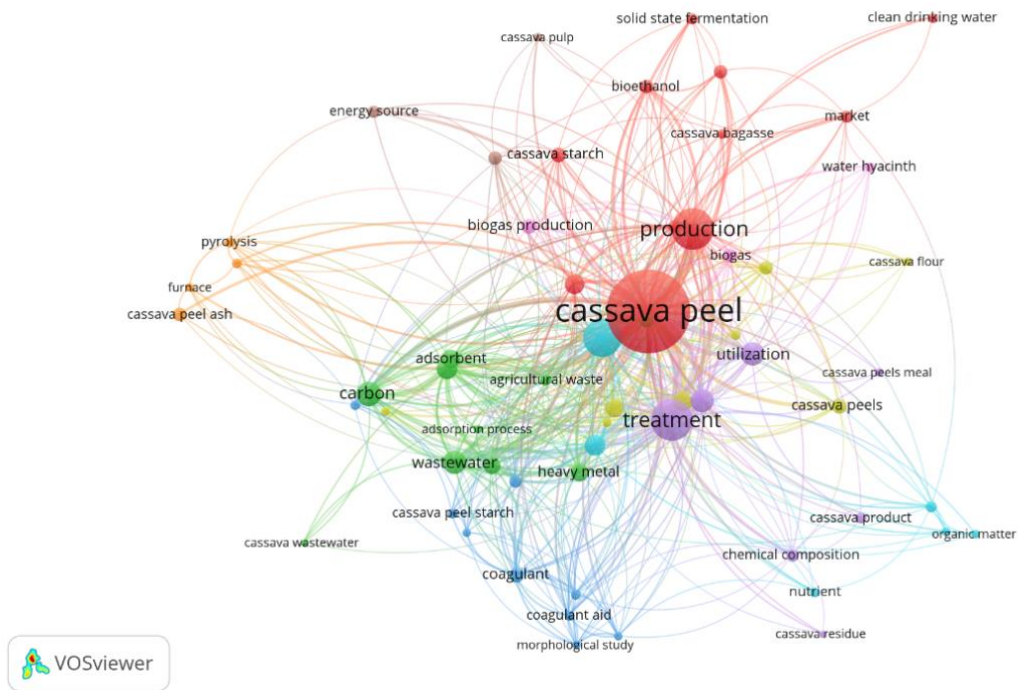


Figure 2. Co-word map network visualization.

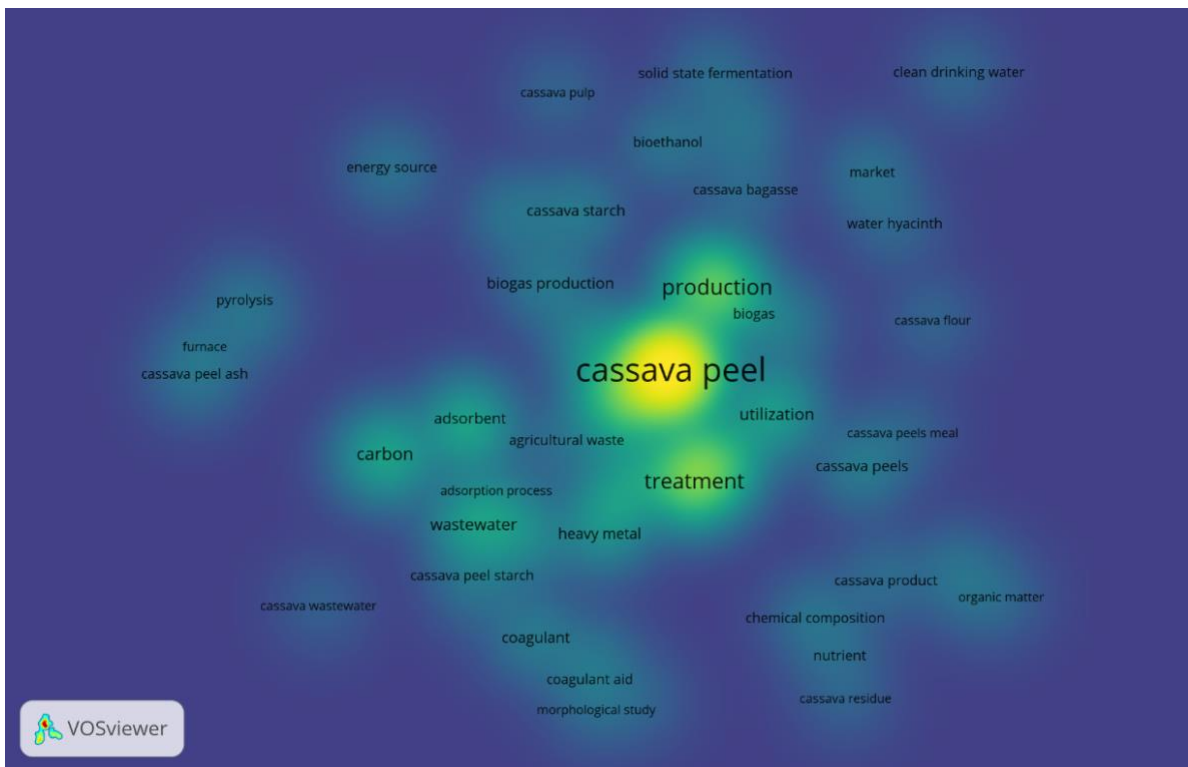
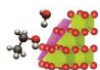


Figure 3. Co-word map density visualization.



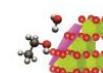
## 2 Result and Discussion

### 2.1 Types of Modified Coagulants

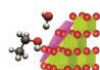
**Table 1** shows some of the CP-based natural coagulant modifications that can be fabricated.

**Table 1.** Methods, materials, and results of the CP-based natural coagulant processing.

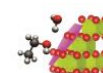
Modification Coagulant	Material	Method	Ref.
CP as Coagulant Aid	CP, distilled water, and binder wax.	<ol style="list-style-type: none"> <li>1. CP was collected</li> <li>2. The raw CP was further processed to separate the sample into three types of samples consisting of CP1 (periderm, cortex and flesh), CP2 (cortex only) and CP3 (meat only)</li> <li>3. Then, the CP sample was washed with water repeatedly to remove the surface dirt.</li> <li>4. Then, the peel is soaked in distilled water for further cleansing for 24 hours</li> <li>5. CP was dried using oven at 60°C for 24 hours to remove moisture.</li> <li>6. The dried peel was then ground into a fine powder using a domestic milled and sieved to obtain the desired particle size &lt;150µm</li> <li>7. The final product is stored in an airtight plastic container for further analysis.</li> </ol>	[19]
Coagulant of cassava peel starch and alum	Samples raw water, cassava peel starch (CPS), deionized water, Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> , HCl and NaOH.	<ol style="list-style-type: none"> <li>1. CP eligible were selected and carefully cleaned using tap water before rinsing with distilled water.</li> <li>2. Then, the periderm layer is scraped off the entire peel using a fruit scraper.</li> <li>3. The peel of the cassava is then cut into pieces about 1 cm long and suspended in distilled water for 2 times its volume</li> <li>4. Then, CP was ground with a milling apparatus for 5 minutes.</li> <li>5. The pulp is then suspended in 10 times the volume of distilled water and stirred for 2 minutes.</li> <li>6. The suspension is then filtered using double-folded gauze.</li> <li>7. The filtrate is allowed to stand for 2 hours to settle before the top liquid is poured and discarded.</li> <li>8. The precipitate was collected and dried in the sun for 12 hours to remove the moisture content.</li> <li>9. Dried CPS is stored in an airtight plastic container for further use optimization studies using Jar test, three optimization studies were carried out consisting of i) alum as coagulant;</li> </ol>	[5]



Modification Coagulant	Material	Method	Ref.
		ii) CPS as coagulant; iii) Alum-CPS combination coagulant.	
Coagulant of cassava peel starch and alum	Cassava peel starch, distilled water, $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ , hydrochloric acid, and sodium hydroxide	<ol style="list-style-type: none"> <li>1. Good quality CP is selected and washed using water repeatedly to remove surface dirt.</li> <li>2. The CP is further processed by removing the outer wood layer using a fruit scrapper.</li> <li>3. The skin is then cut into pieces of 1 cm length and suspended in distilled water twice its volume.</li> <li>4. Then, the suspension was milled for 5 minutes.</li> <li>5. The mixture is filtered through double-folded gauze followed by a sedimentation process of at least 2 hours.</li> <li>6. The top liquid is then removed while the white precipitate is collected and dried in the sun for 12 hours until it reaches a constant weight to ensure all moisture is removed.</li> <li>7. Dried CPS is stored in an airtight plastic container for further experimental use</li> <li>8. Optimization studies using Jar test. There are three optimization studies were carried out consisting of i) alum as coagulant; ii) CPS as coagulant; iii) Alum-CPS combination coagulant.</li> </ol>	[20]
Sustainable Coagulant Alum Water and Cassava Peel Starch (CPS)	Cassava peel starch, distilled water, industrial waste, and $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$	<ol style="list-style-type: none"> <li>1. Collected 20 g of CPS sample</li> <li>2. Samples were peeled, cleaned with water, rinsed with distilled water, and dried</li> <li>3. Next, CPS is mashed using a blender and filtered using a filter cloth into starch slurry</li> <li>4. Then, it is deposited for 30 minutes</li> <li>5. The starch precipitate is separated from the pulp, kept in view, and the cassava peel mass is washed again with distilled water.</li> <li>6. Then, the starch sediment was dried at 70°C for 24 h.</li> <li>7. The dry peel was grounded again into a fine powder using an milling apparatus and then sieved to obtain a size between 400 – 800 mesh</li> <li>8. Prepared alum solutions with various concentrations for coagulation tests</li> <li>9. CPS coagulant stock solution was prepared by dissolving 200 g of CPS powder in 1 L of distilled water.</li> </ol>	[4]



Modification Coagulant	Material	Method	Ref.
		<ol style="list-style-type: none"> <li>10. The suspension is mixed vigorously using a magnetic stirrer for one hour</li> <li>11. Fresh suspensions were prepared daily and stored in closed flasks at room temperature (10-19 °C) for one week.</li> <li>12. The dose required for each flocculation run is designed from the stock solution.</li> <li>13. Preparation of stock solution for CPS by adding 200 g of CPS powder to 1L of distilled water to produce a 0.1 percent suspension (Concentrate).</li> <li>14. A mixture consisting of alum and CPS was prepared in a ratio (0:20 to 100:0 CPS: alum).</li> <li>15. Coagulation-flocculation experiments were carried out using a conventional jar test apparatus (JLT 6, 6 position tube tester)</li> </ol>	
<p>Modified double coagulant cassava peel starch synthetic lemongrass extract with alum.</p>	<p>Cassava peel, lemongrass extract, distilled water HCl, NaHCO<sub>3</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.18H<sub>2</sub>O, etanol and methanol 95%</p>	<ol style="list-style-type: none"> <li>1. CP are obtained from local cassava chip factories.</li> <li>2. Good quality CP is selected and cleaned with tap water to remove surface dirt.</li> <li>3. The outer layer is removed manually. 100g of peeled skin was then suspended in 200 ml of distilled water and mashed using a household milling for 5 minutes.</li> <li>4. Next, 2000 mL of distilled water was added to the suspension and stirred for 2 minutes.</li> <li>5. Then, the suspension was filtered using double-folded gauze and the filtrate was allowed to stand for 2 hours to be precipitated.</li> <li>6. Then, the sediment is collected and dried in the sun for at least 24 hours to remove the moisture content.</li> <li>7. Dried CPS was stored in an airtight glass container for further experimental use.</li> <li>8. 20 g of dry CPS were suspended in 100 mL of 100% ethanol extract v/v lemongrass and stirred at 150 rpm for 1 hour at 25°C</li> <li>9. Then, the reaction was started by adding 1 mL of concentrated HCl (36%) and left at 25 °C for 24 hours with continuous stirring at 150 rpm.</li> <li>10. The reaction was stopped by adding 14 mL of 1 M NaHCO<sub>3</sub> The solution was cooled in an ice bath for 5 minutes</li> <li>11. Then, centrifuged at 4000 rpm for 5 minutes.</li> </ol>	<p>[21]</p>



Modification Coagulant	Material	Method	Ref.
		12. Furthermore, the sediment is dried in the sun until it reaches a constant weight. 13. The modified cassava starch (MCPS) was finally stored as a solid powder for further analysis.	
Cassava peel coagulant	Samples of raw water, cassava peel, distilled water, $\text{Al}(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$	1. CP is cleaned using tap water before rinsing with distilled water. 2. After that, the periderm layer is scraped from the entire skin using a fruit scraper. 3. The peel is then cut into pieces about 1 cm long and mashed. 4. The pulp is then suspended in 10 times the volume of distilled water and stirred for 2 minutes. 5. The suspension is then filtered using double-folded gauze. 6. The filtrate is allowed to stand for 2 hours to settle before the top liquid is poured and discarded. 7. The precipitate was collected and dried in an oven at $60^\circ\text{C}$ for 24 hours to remove the moisture content. 8. Dried CPS is stored in an airtight plastic container for use further. 9. Three sets of jar tests were performed, optimization was investigated the effect of coagulant dose: alum (5, 10, 15, 20, 25, 30 mg/L), CP (100, 200, 300, 400, 500, 600mg/L), alum + CP (100%:0%, 70%:30%, 50%:50%, 30%:70%, 0%:100%). The pH and settling time were kept constant at pH 7 and 60 min.	[19]

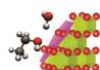
## 2.2 Cassava Peel Coagulants Characterization

In this study, before further analysis, it is necessary to carry out a characterization test to determine the structure and surface morphology of the cassava peel. Physicochemical analysis was carried out with several characterizations as follows: i) morphology characterization (using Scanning Electron Microscope (SEM) and Field Emission Scanning Electron Microscopy (FE-SEM instruments); ii) functional group analysis (using Fourier Transformed Infrared (FTIR)

instrument); iii) chemical composition analysis (using X-Ray Fluorescent (XRF) instrument); and iv) molecular structure analysis (using Proton Nuclear Magnetic Resonance (NMR proton) instrument). The following is a cassava peel characterization test [19].

### 2.2.1 Field Emission Scanning Electron Microscopy (FE-SEM) and Scanning Electron Microscope (SEM)

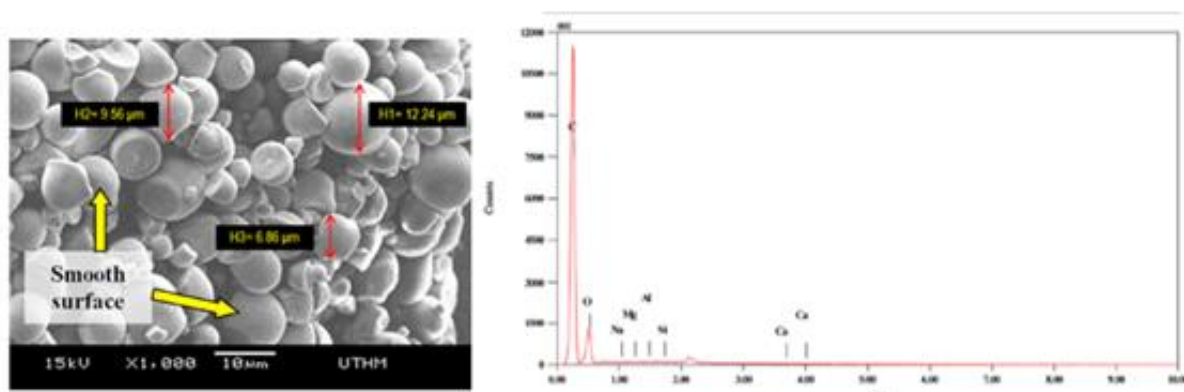
The morphology and distribution of the original surface elements of alum, native CPS, and



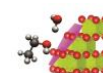


MCPS are presented in **Figures 4-6**, respectively. **Figure 4** is the surface morphology and EDX spectrum of alum. As can be seen, the solid particles form clumps made of crystals of various sizes. The average size of alum clumps is about 40-60  $\mu\text{m}$ , similar to the observations reported by [6]. The distribution of surface elements reflected by EDX spectrometry confirmed the absence of impurities in the alum sample used as the main coagulant in this study. Both starches exhibit polygonal and spherical starch granules with clear ridges that appear to mark sites, where the granules are in close contact with one another [31][32]. In addition, the original CPS starch granules were observed to be finer with no evidence of cracking due to the low level of damage during the starch isolation process [6]. EDX analysis performed on native CPS only identified the presence of prominent C and O peaks, confirming the assumption that pure starch granules consist of carbohydrate monomers (see **Figure 5**). Meanwhile, microscopic analysis of MCPS showed a rough surface and wave-like folds (see **Figure 6**), which might be due to the penetration of HCl through the channels and cavities of starch granules during the modification process as observed by Palacios-Fonseca et al. [33] and Van Hung et al. [34]. However, it was observed that the modification of the method did

not greatly affect the morphological characteristics of the starch granules, where no depressions, cracks, or hollow grains were observed. Apparently, as observed in the EDX spectrum of the MCPS presented in **Figure 6**, a significant percentage of the new elements Na, Cl, K, and Ca were detected. The appearance of these elements in MCPS indicates the occurrence of modification and proves that lemongrass extract and HCl were grafted onto CPS. Starch modification using citronella extract has superimposed the characteristic surface elements of MCPS, which indicates an increase in the possible antibacterial properties and metal binding ability in various parts of the coagulant. Increased levels of chlorine have the potential to increase the bactericidal effect of the material as reported by Maharjan et al. [35]. Meanwhile, Ca, K, and Na are natural elements in materials of plant origin, can be removed as heavy metal substitutions during the adsorption process through an ion exchange mechanism [27]. These cationic elements also contribute to enhancing the charge attraction properties of the coagulant not only to destabilize suspended particles but are also effective in removing *E. coli* which is known to bear negative surface charge removal via electrochemical disinfection mechanism [36].



**Figure 4.** SEM-EDX micrograph of alum ( $\times 1000$ ) [6].



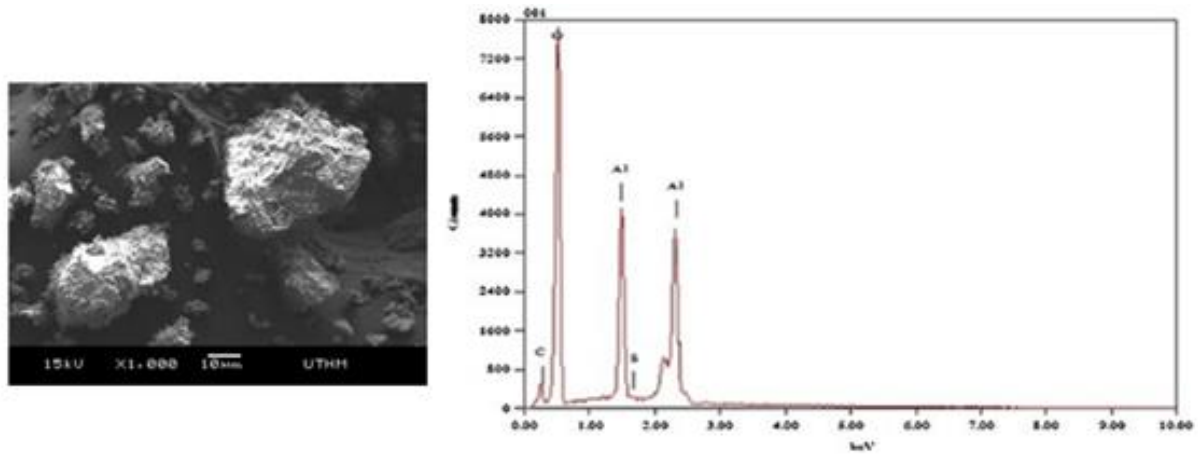


Figure 5. SEM-EDX micrograph of native CPS ( $\times 1000$ ) [6].

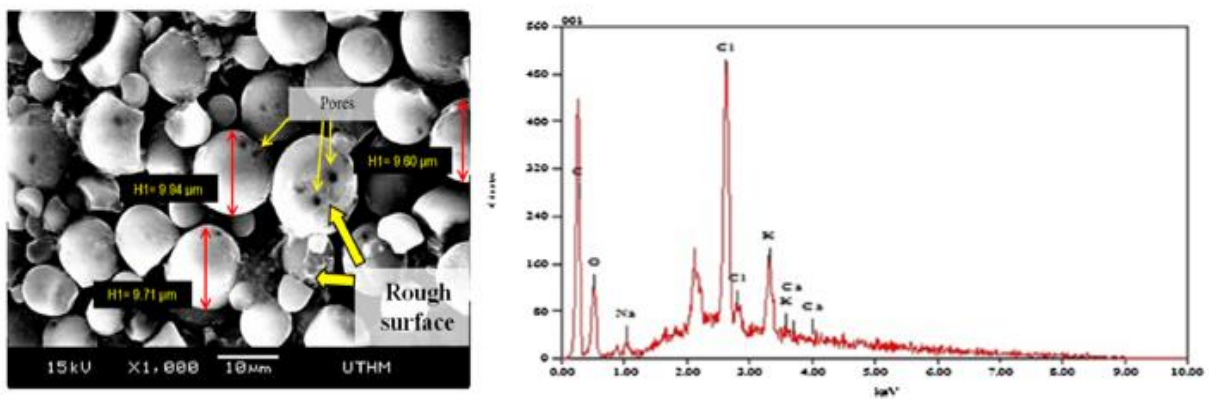
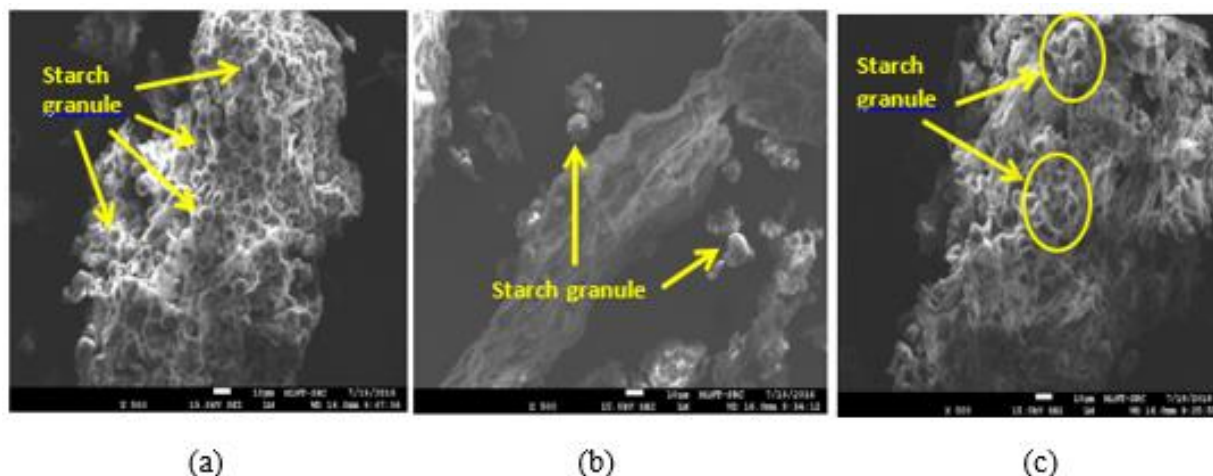


Figure 6. SEM-EDX micrograph of MCPS ( $\times 1000$ ) [6].

Figure 7 is the result of FESEM analysis of CP (for CP1 (periderm, cortex and flesh), CP2 (cortex only) and CP3 (meat only)). This analysis was conducted to determine the surface morphology of the CP sample, FE-SEM micrographs were observed using a JEOL JSM-7600F Field Emission Scanning Electron Microscope (USA). Based on SEM micrographs at 500x magnification, it is seen that the CP surface has non-porous and heterogeneous characteristics. Similar observations were

reported by another study on SEM analysis of cassava peels [7]. The finely bound starch granules and globular in shape were observed mostly covering the entire surface of CP1 and appeared to be clustered in the CP3 sample. Meanwhile, free starch granules that were not bound were observed at CP2. The CP1 sample consisted of all CP waste consisting of periderm, cortex, and flesh layers which may be the cause of the appearance of many starch granules on its surface.





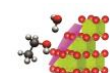
**Figure 7.** FE-SEM micrograph of CP1 (a), CP2 (b), and CP3 (c) [19]

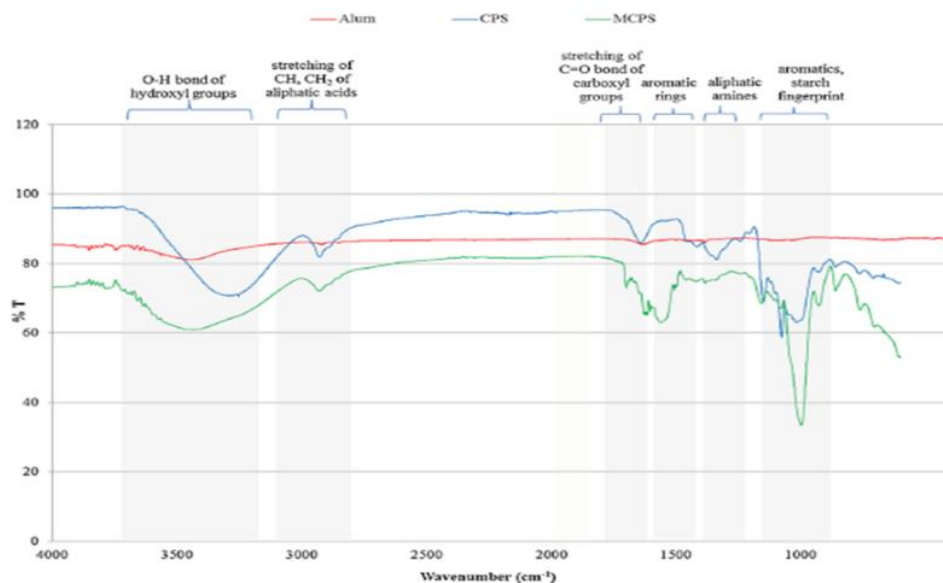
### 2.2.2 Fourier Transform Infrared (FTIR) Spectroscopy

**Figure 8** shows FTIR spectra of alum, native CPS, and MCPS. The FTIR spectra of the alum samples indicated that the compound was hydrate due to the observed wide absorption band between 3500 and 3200  $\text{cm}^{-1}$  caused by hydroxide stretching vibrations and the absorption band around 1620  $\text{cm}^{-1}$  corresponds to the OH bending vibration mode, which confirms the presence of free molecules and water, respectively. The peak at the wavenumber of 1075  $\text{cm}^{-1}$  can be attributed to the asymmetric stretching of the free sulfate anion. The bands identified for alum are similar to the findings reported in the literature [25].

Meanwhile, compared to the spectrum for CPS, changes in the molecular level of MCPS as a result of the modification process were proven from the FTIR analysis. The FTIR spectra confirmed the change in the chemical group of starch, which was determined by the band shift, as

well as the formation of minor and major peaks. The change in peak position can be attributed to the change in counter ion associated with the carboxylate and hydroxylate anions which mostly belong to the carbonyl and hydroxyl groups present in the starch molecule [26]. Important changes are seen in the broad band at 3400-3600  $\text{cm}^{-1}$ , which may be due to a complexation reaction involving the ionized OH group of the free hydroxyl group and bound in the carboxylic acid. These groups are commonly found in polymeric compounds such as alcohols and phenols of carbohydrate polymers [27]. Since CPS mainly consists of glucose monomers, it is well demonstrated from the FTIR spectrum that the carboxyl and hydroxyl groups are present in abundance. These groups available in the biopolymer can serve as proton donors; hence, deprotonated hydroxyl and carboxyl groups may be involved in the formation of alum starch complexes [28].





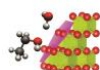
**Figure 8.** FTIR spectra of alum, native CPS, and MCPS.

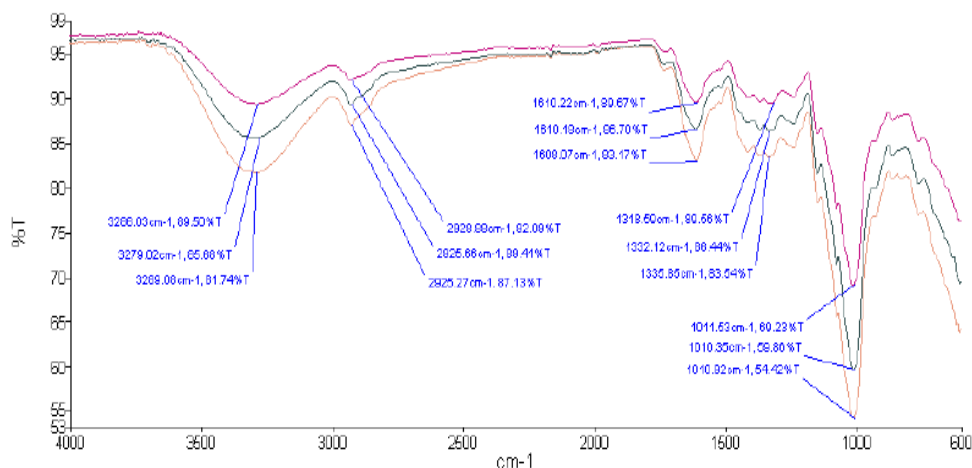
Changes in the region between 2900 and 3000  $\text{cm}^{-1}$  may indicate an ion exchange reaction driven by symmetric proton substitution or asymmetric CH bonds and symmetric stretching vibrations of  $\text{CH}_2$  belonging to aliphatic acids. Meanwhile, the peak that appears between 1750 and 1680  $\text{cm}^{-1}$  shows the stretching vibration of the CO bond due to the non-ionic carboxyl group ( $\text{COOH}$ ,  $\text{COOCH}_3$ ) and also the hydrogen bond between the carboxylic acid and the ester group [29].

Peak changes were also observed between wavenumbers 1400 and 1200  $\text{cm}^{-1}$ , reflecting the stretch vibrations of the symmetrical or asymmetrical ionic carboxylate groups ( $\text{COO}^-$ ) of starch, and may indicate that an ion exchange process has occurred [30]. This phenomenon can be attributed to the observations obtained from the EDX analysis, in which some elements such as Na, Cl, K, and Ca were newly added in the MCPS

samples and might be integrated in the starch framework via an ion exchange mechanism. Moreover, a characteristic change was also noted at 1016  $\text{cm}^{-1}$ , wherein the peak intensity of the MCPS increased significantly, thus forming a deep peak. Several studies have linked the band in the area between 1016 and 600  $\text{cm}^{-1}$  with the strain characteristics of the terpene component CO, which may be derived from the active components of lemongrass such as citral, geraniol, and linalool [29].

**Figure 9** shows the results of the FTIR analysis of the CP coagulant for CP1 (periderm, cortex and flesh), CP2 (cortex only), and CP3 (meat only). FTIR was performed to identify the surface functional groups of the original CP samples. Adsorption bands were recorded at characteristic wave numbers between 600  $\text{cm}^{-1}$  and 4000  $\text{cm}^{-1}$ .





**Figure 9.** FTIR Spectrum of CP1, CP2 and CP3

The peaks that appear in the FTIR spectrum are assigned to various chemical groups and bonds according to their respective wavenumbers ( $\text{cm}^{-1}$ ). The observed broad band between  $3500 - 3200 \text{ cm}^{-1}$  can be attributed to the OH group of the free hydroxyl group and the bound OH group in polymeric compounds such as alcohols, phenols and carboxylic acids presented in pectin, cellulose and lignin in CP. These bioactive components have been found to have antimicrobial properties. The peak at wave numbers between  $3000 - 2850 \text{ cm}^{-1}$  can indicate a symmetrical or asymmetrical CH stretch and CH ( $\text{sp}^2$ ), of aliphatic acids. Furthermore, the characteristic peak appears between  $1750 - 1680 \text{ cm}^{-1}$  demonstrating the stretching vibration of the C=O carboxyl group. Meanwhile, the peak observed between  $1375 - 1300 \text{ cm}^{-1}$  reflects the stretching vibration of the pectin ion carboxylate group. While the inner peak between  $1300 - 1000 \text{ cm}^{-1}$  indicates stretching of CO from  $-\text{COOH}$  [19].

Since the cassava peel is mainly composed of lignin and cellulose, it is well demonstrated from the FTIR spectrum of each sample that carboxyl and hydroxyl groups are present in abundance especially in sample CP1. The groups that make up this biopolymer can serve as proton donors; hence the deprotonated hydroxyl and carboxyl groups may be involved in the formation of alum starch complexes. The insoluble alum starch salts can initially form micro flocs (coagulation) which gradually grow into macro flocs (flocculation).

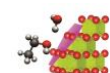
Over time, macro flocs settle and increase water clarity [20].

Awang and Abdul-Aziz [22] showed that carboxyl (C=O), hydroxyl (O-H) and amino or amide ( $\text{NH}_2$ ) groups as well as hydrogen bonds, which are the preferred groups for the flocculation process. The deprotonated carboxyl ( $-\text{C}=\text{O}-$ ) group can also function as an ion bridge or binding site for divalent metal cations ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , etc.) on the surface of wastewater particles to induce coagulation activity.

### 2.2.3 X-Ray Fluorescent (XRF) Spectrometry

All CP samples were also subjected to XRF analysis to determine the elemental composition of the samples using a Bruker AXS S4 Pioneer XRF spectrometer [7]. **Table 2** shows percentage of element composition of in various cassava peel, especially for CP1, CP2, and CP3. The elements present in CP that provide coagulant properties are  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ . These two elements are expected to give CP the ability to assist the coagulation process through the deposition of particles in raw water.

Standard alum consists of 17-18 wt%  $\text{Al}_2\text{O}_3$  and 0.4-0.7 wt.  $\text{Fe}_2\text{O}_3$ . In addition, the presence of  $\text{SiO}_2$  and  $\text{CaO}$  in the CP might aid in the addition of weight and particle size in raw water as in the case of active silicates as flocculation aids [19]. In addition,  $\text{SiO}_2$  is also found in CP. The same observation was reported in the rice husk characterization study[23]. This study proposes that the elements that may be responsible for the



absorption are carbon and silica. Rao et al. [24] suggested that when the Si-OH bond is present it will also act as a cation exchange center. This could be due to the substitution of metal ions at the

site resulting in a net negative charge which allows the adsorption of cations. Based on **Table 2**, the content of each element of CP1, CP2, and CP3 is not the same.

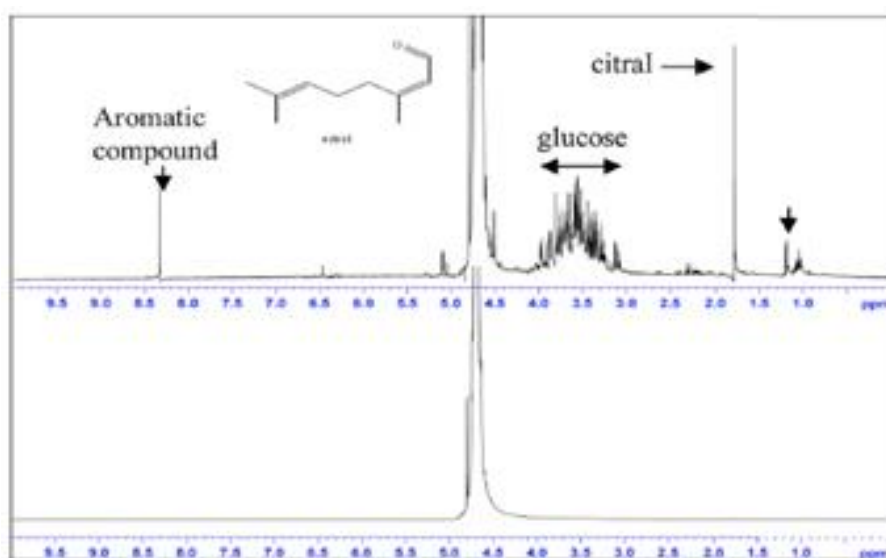
**Table 2.** Composition of CP (adopted from reference [19])

Element	Weight (%)		
	CP1	CP2	CP3
C	0.10	0.10	0.10
K <sub>2</sub> O	5.48	6.18	5.83
CaO	4.20	3.90	3.48
Fe <sub>2</sub> O <sub>3</sub>	1.54	0.33	0.41
SiO <sub>2</sub>	0.87	0.23	0.40
SO <sub>3</sub>	0.87	0.94	0.94
Al <sub>2</sub> O <sub>3</sub>	0,74	-	0,26
P <sub>2</sub> O <sub>5</sub>	-	-	0,42

#### 2.2.4 <sup>1</sup>H NMR Analysis

The parameters of the chemical composition of starch were then measured by the H NMR technique and the spectrum is presented in **Figure 10**. A significant increase of the new peak was observed in the MCPS samples due to chemical substitution involving anomeric protons in -(1,4) and -(1,6) glycosidic linkages with plant secondary metabolites [37]. Modified starch revealed new peaks at 1.09 and 1.20 ppm, which were re-flected for CH<sub>3</sub> in the alcohol group.

Meanwhile, intense peaks appeared at 1.78 ppm associated with active lemongrass groups including neral and geranial, which is an isomer of citral (3,7-dimethyl-2,6-octadienal). Signals indicating carbohydrate monomers including  $\alpha$ -glucose and  $\beta$ -glucose were also observed as broad peaks at 3-4 ppm for the MCPS samples[38]. Observations based on NMR signals have proven the success of perfusion of the active compounds of lemongrass extract into native starch.



**Figure 10.** <sup>1</sup>H NMR spectra of (a) MCPS and (b) native CPS.



### 2.3 Parameter Optimization in Performance of Modified Coagulant

Choosing the most efficient material for a specific system can be difficult. As previous mention, coagulant materials used include inorganic salts, polymeric coagulants, organic polyelectrolytes, and composite inorganic-organic coagulants, and cost and storage are important considerations in addition to optimizing removal efficiency. This section discusses some of the factors to consider when choosing a coagulant system.

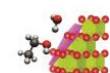
#### 2.3.1 pH Optimization

**Table 3** shows the variation of certain pH conditions to determine the optimal performance of the cassava peel-based coagulant. pH is the main parameter in the coagulation and flocculation process, because it can affect the surface charge of the coagulant and the stability of the suspension. In Asharuddin's research, 2018 [39], it was observed changes in pH for TSS removal until optimum conditions were obtained at pH 7 for the cassava peel starch coagulant type and at pH 9 for the dual coagulant Alum-Cassava Peel Starch. In an acidic environment, the lone pair of nitrogen electrons from the amino group on the granular surface of starch becomes protonated and becomes positively charged while the carboxyl group becomes neutral [40]. Meanwhile in Ahsaruddin [6], using Cassava peel starch modified with lemongrass extract as a dual coagulant in testing TSS removal showed that the optimum conditions for pH parameters occurred in neutral conditions. Under the optimum pH conditions when alum is used in combination with starch coagulant, hydroxoaluminium cations (monomers and polymers) coexist with aluminum hydroxide precipitates [41]. Metal cations can neutralize the negative charge of pollutants through surface adsorption, while metal cation polymers can increase the binding between pollutant particles effectively through the sedimentation process.

**Table 3.** Variation of pH to determine the optimal performance of of cassava peel-based coagulant at certain pH conditions.

Coagulant	pH	References
Cassava Peel Starch	2, 3, 4, 5, 6, <b>7</b> , 8, 9, 10, 11	[5]
	2, 3, 4, 5, 6, 7, 8, 9, 10, 11	[20]
	2, 3, 4, <b>5, 6, 7</b> , 8, 9, 10, 11	[4]
Alum – Cassava Peel Starch	2, 3, 4, 5, 6, 7, 8, <b>9</b> , 10, 11	[5]
	2, 3, 4, 5, 6, 7, 8, <b>9</b> , 10, 11	[20]
	2, 3, 4, 5, 6, <b>7</b> , 8, 9, 10, 11	[6]
Alum – Modified Cassava peel with lemongrass extract	2, 3, 4, 5, 6, 7, <b>8</b> , 9, 10, 11	[5]
	2, 3, 4, 5, 6, 7, 8, 9, 10, 11	[6]

Note: The parameter values in bold are the best conditions in the water treatment process



In a different study by Asharuddin [20], it was shown that the turbidity removal by the dual coagulant system Alum-cassava peel starch was optimum at an initial pH of 9, while for cassava peel starch alone was optimum at pH 2. Saxena [42] determined that the solubility of alum depending on pH and optimum in the pH range of 5 - 7.5, and if the pH is outside this range, the coagulation activity using alum salt will be inhibited. Abundant natural coagulants with active anionic components can serve as alkalinity reservoirs and facilitate acid neutralization resulting from hydrolysis of aluminum ions [43]. In the natural environment, the hydrolysis of aluminum ions causes a reduction in pH in the treated water. Therefore, hydrated lime is often used in water treatment to neutralize the pH of treated water. Therefore, as obtained from the current study, the initial pH of 9 required for optimal removal of turbidity by Alum-cassava peel starch can be considered as an advantage that creates a sufficient environment for floc formation as well as producing a neutral pH of the treated water.

### 2.3.2 Coagulant Dose Optimization

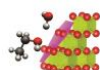
**Table 4** shows the variation in dosage to determine the optimal performance of cassava peel-based coagulant. In Asharuddin's research [39], increasing alum dose can cause more suspended particles to be entangled in hydroxide precipitates to increase TSS removal. The same effect occurs when the dose of cassava peel starch is increased, but in excess doses it can cause a decrease in TSS removal because the surface of the polymer chain is excess saturation, which triggers particle stabilization. Meanwhile, Asharuddin's research in 2019 showed the optimum dose for the dual coagulant system Alum-Modified Cassava peel with lemongrass extract, namely at a dose ratio of 7.5 mg/L: 50 mg/L. The optimum condition is obtained when the electro kinetic potential of the colloidal medium is reduced by alum through a charge neutralization process. This occurs because the modified starch has a lower repulsion between the anionic starch species and colloidal particles, so that it gradually develops into larger flocs and then settles.

**Table 4.** Variations in dosage to determine the optimal performance of of cassava peel-based coagulant under certain coagulant dosage conditions.

Coagulant	Coagulant doses (mg/l)	References
Cassava Peel Starch	100, <b>200</b> , 300, 400, 500, 600	[5]
	100, <b>200</b> , 300, 400, 500, 600	[20]
Alum – Cassava Peel Starch	100%:0%, <b>70%:30%</b> , 50%:50%, 30%:70%, 0%:100%	[5]
	100%:0%, 70%:30%, <b>50%:50%</b> , 30%:70%, 0%:100%	[20]
	15:100, 15:0, 11.25:25, <b>7.5:50</b> , 3.75:75, 0:100	[6]
Alum – Modified Cassava peel with lemongrass extract	100%:0%, 70%:30%, 50%:50%, 30%:70%, <b>20%:80%</b> , 0%:100%	[4]
	15:100, 15:0, 11.25:25, <b>7.5:50</b> , 3.75:75, 0:100	[6]

Note: The parameter values in bold are the best conditions in the water treatment process

Another Asharuddin study [20] concluded that the optimum dose of cassava peel starch as a coagulant was 200 mg/l, while the alum-cassava peel dual coagulant system was optimum at the ratio 50%:50% (7.5 mg/L : 100 mg/L) with a turbidity removal value of 90.32%. The value of turbidity removal in cassava peel starch as a coagulant was obtained because the content of amylopectin which can be the main agent as a





bridge forming between particles contained in cassava peel starch with a fairly high concentration. As a result, it can increase the colloid mass and accelerate the precipitation process [44]. Meanwhile, the value of turbidity removal in the dual coagulant system occurs because of the interaction between the cationic charge of  $Al^{3+}$  and the anionic charge of the starch to produce an insoluble aluminum salt. This salt is used to initiate the formation of micro-flocs at an early stage, then gradually form larger-sized flocs [19].

### 2.3.3 Settling time optimization

**Table 5** shows variation of settling time to determine the optimal performance of cassava peel-based coagulant. The most optimal settling time for both types of coagulants in Asharuddin's study [39] was 60 minutes and 30 minutes, respectively. The rate of deposition of the Alum-Cassava Peel starch coagulant was faster due to the interaction between the high molecular weight of the cassava peel starch polymer chain and the initial floc formed by the addition of alum. In another Asharuddin study [6], the optimum condition for settling time in the dual coagulant system of Alum – Modified Cassava peel with lemongrass extract was 10 minutes. The high deposition rate was caused by the long chain structure of the starch with the addition of additional metabolites from the lemongrass extract which contributed to the high molecular weight. This gives rise to a phenomenon where the opportunity for dual coagulants to interact with colloidal particles is greater, and leads to the formation of larger flocs and a faster deposition process.

In Asharuddin's research [20], the optimum conditions for setting time parameters for cassava peel starch and dual coagulant Alum-cassava peel starch were 60 and 30 minutes, respectively, based on turbidity removal values (85.90% and 91.47%). In addition, in the dual coagulant system Alum-Cassava Peel Starch resulted in a significantly higher turbidity removal value compared to the coagulant only Cassava Peel Starch. A study explained that the high speed deposition obtained in the dual coagulant system of Alum-cassava peel

starch was due to the long chain structure of the starch which made it high in molecular weight. This characteristic allows the alum-starch complex to interact with more colloidal particles at the same time leading to the formation of larger and denser flocs which can settle more quickly [45].

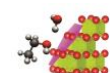
**Table 5.** Variation of settling time to determine the optimal performance of of cassava peel-based coagulant at certain settling time conditions.

Coagulant	Settling time (min)	References
Cassava Peel	10, 20, 30, <b>60</b> , 90, 120	[5]
	10, 20, 30, <b>60</b> , 90, 120	[20]
Alum – Cassava Peel Starch	10, 20, 30, <b>60</b> , 90, 120	[4]
	10, 20, <b>30</b> , 60, 90, 120	[5]
Alum – Cassava Peel Starch	10, 20, <b>30</b> , 60, 90, 120	[20]
	10, 20, <b>30</b> , 60, 90, 120	[6]
Alum – Modified Cassava peel with lemongrass extract	<b>10</b> , 20, 30, 60, 90, 120	[6]

Note: The parameter values in bold are the best conditions in the water treatment process

### 2.3.4 Slow mixing speed optimization

**Table 6** shows variation of slow mixing speed to determine the optimal performance of cassava peel-based coagulant at certain time conditions. The effect of slow mixing speed was varied in the speed range of 1 to 50 rpm in Asharuddin's study, 2018 [20]. This study showed that the highest turbidity removal value of 90.01% was obtained in the dual coagulant system of Alum-cassava peel starch at 10 rpm, while the slow mixing speed for Cassava Peel Starch coagulant was optimum at 20 rpm. From the observations, it can be seen that when the speed is higher than the optimum condition (> 20 rpm) it will cause a statistically significant decrease in the



value of turbidity removal ( $P < 0.05$ ). This is due to the effectiveness of the disruptive force, resulting in floc breaking and less particle collision in the shear field [46].

**Table 6.** Variation of slow mixing speed to determine the optimal performance of cassava peel at certain settling time conditions.

Coagulant	Mixing speed (rpm)	References
Cassava Peel Starch	0, 10, <b>20</b> , 30, 40, 50	[20]
Alum – Cassava Peel Starch	0, <b>10</b> , 20, 30, 40, 50	[20]

Note: The parameter values in bold are the best conditions in the water treatment process

In the early stages of the coagulation and flocculation process, fast mixing is carried out to

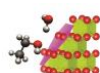
induce uniform dispersion by the coagulant in suspension so that a destabilization reaction occurs and primary floc is formed [43]. In the slow mixing stage, the floc particles are allowed to collide with each other to form aggregates with an increasing size before the sedimentation process is carried out. In the slow mixing process it can affect the value of turbidity removal because in this process it encourages the formation of aggregates, the association of metal cations to functional groups and ligands, and the binding of organic matter with coagulants [47].

#### 2.4 Cassava Peel Coagulant and Adsorbent Application for Various Wastewater Treatment

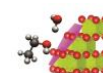
The top-down fabricated particles from cassava peels can be applied in various fields, one of which is as a coagulant material that is applied to wastewater treatment. **Table 7** summarizes the application of cassava peels fabricated particles as coagulant in wastewater treatment.

**Table 7.** Summary of application of top-down fabricated particles from cassava peel as a coagulant and adsorbent for wastewater treatment.

Coagulant/Adsorbent based Cassava Peel and Supporting Materials	Wastewater Type	Results	Ref.
Coagulant Type: Dual coagulant (cassava peel + alum)  Supporting Materials: • Cassava peel • Aluminum sulfate ( $Al_2(SO_4)_3 \cdot 18H_2O$ )	Raw water from Sembrong dam, Malaysia	<ul style="list-style-type: none"> <li>The results obtained from the optimization study confirmed the positive potential of the cassava peel-based coagulant.</li> <li>Treatment using alum-cassava peel combination has achieved the removal of turbidity up to 91.47% using optimal conditions at pH 9, alum dose of 7.5mg/L, assisted with 100 mg/L cassava peel dose, 200 rpm rapid mixing for 1 minute, continued 10 rpm for 10 minutes, and settling time for 30 minutes.</li> <li>Compared to treatment with alum alone, the addition of 100 mg/L cassava peel has succeeded in reducing 50% of alum dose with the highest percentage of turbidity removal.</li> </ul>	[20]
Coagulant Type: Modified Coagulant Lemongrass Extract  Supporting Materials: • Cassava peel • Lemongrass Extract	Raw Water, Malaysia	<ul style="list-style-type: none"> <li>The integration of the active group from the lemongrass extract into the cassava peel starch was successfully carried out.</li> <li>The results of this study provide a promising potential for manipulating natural precursors as an alternative to modified starch to improve the properties</li> </ul>	[21]



<ul style="list-style-type: none"> <li>• Chloride Acid (HCl)</li> <li>• Sodium Bicarbonate (NaHCO<sub>3</sub>).</li> </ul>		of starch as flocculants and disinfectants for water treatment.
Coagulant Type: Aid Coagulant  Supporting Materials: <ul style="list-style-type: none"> <li>• Cassava peel</li> <li>• Aluminium sulfate</li> </ul>	Institutional wastewater	<ul style="list-style-type: none"> <li>• The application of cassava peel in institutional wastewater treatment shows that it can be used effectively as a natural coagulant to replace chemical coagulants in removing cloudy particles in wastewater.</li> <li>• Cassava peel used for testing using the jar test method was 30mg/L and 40:60 mg/L which succeeded in removing water turbidity up to 81% of the initial turbidity of raw water.</li> </ul>
Adsorben Type: Cassava Peel Adsorbent Modified Sodium Dodecyl Sulphate  Supporting Materials: <ul style="list-style-type: none"> <li>• Cassava peel</li> <li>• Sodium dodecyl sulphate (SDS)</li> <li>• Phosphoric Acid</li> </ul>	Metilen Blue (MB) in aqueous solution	<ul style="list-style-type: none"> <li>• MB adsorption gradually increases with increasing surfactant mass, up to a maximum adsorption capacity at 15 mg surfactant mass.</li> <li>• In this case, the surface of the biochar is covered by the negative charge of the surfactant molecules, thus there is an increases in the adsorption of MB to the surface of the biochar at the highest surfactant mass.</li> </ul>
Adsorbent Type: Cassava Peel modified HNO <sub>3</sub> Biosorbent  Supporting Materials: <ul style="list-style-type: none"> <li>• Cassava peel</li> <li>• HNO<sub>3</sub></li> </ul>	Chromium (VI) waste in electroplating waste	<ul style="list-style-type: none"> <li>• The optimum mass of cassava peel activated biosorbent with 1.5 M HNO<sub>3</sub> was 0.3 gram and the optimum contact time was 10 minutes. Furthermore, the mass and optimum contact time of cassava peel activated with 1.5 M HNO<sub>3</sub> biosorbent can be applied to electroplating waste.</li> <li>• The average percentage of chromium (VI) adsorbed on the electroplating waste was 61.72% with the optimum mass treatment and contact time of cassava peel activated with 1.5 M HNO<sub>3</sub>.</li> <li>• The percentage of chromium (VI) removal that was adsorbed on average was 61.72%.</li> </ul>
Adsorbent Type: Immobilized Cassava Peel Adsorbent  Supporting Materials: <ul style="list-style-type: none"> <li>• Cassava peel</li> <li>• HNO<sub>3</sub></li> <li>• Thioglycollic acid</li> </ul>	Heavy Metal Waste	<ul style="list-style-type: none"> <li>• The results of the biosorption-desorption show that the biosorbent can be used repeatedly without significant loss of adsorption capacity which reflects its feasibility for commercial applications.</li> <li>• In addition, its easy availability and low cost make immobilized cassava peel biomass as an attractive biosorbent option</li> </ul>
Adsorbent Type: Cassava Peel Adsorbent  Supporting Materials: <ul style="list-style-type: none"> <li>• Cassava Peel</li> </ul>	Ni-sorption	<ul style="list-style-type: none"> <li>• The maximum absorption (qm) of Ni by cassava peel was 57 mg/g at pH 4.5.</li> <li>• The Sips model provides the best representation for the adsorption equilibrium data.</li> </ul>



<p>Adsorbent Type: Thioglycolic acid modified Cassava Peel adsorbent</p>	<p>Heavy Metal (V<sup>3+</sup>, Co<sup>2+</sup>, and Cr<sup>3+</sup>)</p>	<ul style="list-style-type: none"> <li>• Eight stages of Co<sup>2+</sup> removal are required [50] to meet the target limit of 0.5 mg/L in waste disposal.</li> <li>• Cr<sup>3+</sup> adsorption requires six steps to obtain a target limit of 0.1 mg/L.</li> <li>• For V<sup>3+</sup>, requires 4 stages with a target limit of 0.2 mg/L.</li> <li>• In general, the biosorption efficiency of cassava peels increases with the order of Co<sup>2+</sup> &lt; Cr<sup>3+</sup> &lt; V<sup>3+</sup>.</li> </ul>
<p>Supporting Materials:</p>		
<ul style="list-style-type: none"> <li>• Cassava Peel</li> <li>• Nitric acid</li> <li>• Thioglycolic Acid</li> </ul>		
<p>Adsorbent Type: Cassava Peel-based Activated Carbon Adsorbent</p>	<p>Malachite green dye (C<sub>52</sub>H<sub>54</sub>N<sub>4</sub>O<sub>12</sub> )</p>	<ul style="list-style-type: none"> <li>• The percentage of adsorption efficiency [51] of the adsorbent treated with silver nitrate, sulfuric acid, and raw cassava peel carbon decreased with increasing concentration of the initial dye in the solution.</li> <li>• For cassava peels treated with silver nitrate and sulfuric acid, equilibrium was reached only after 60 and 90 minutes, respectively.</li> <li>• Adsorption increased from 48.9 to 94.1%, because the adsorbent dose treated with silver nitric acid increased from 0.2 g to 1.0 g/150 mL at the equilibrium time of 120 minutes.</li> <li>• For cassava peel treated with sulfuric acid, adsorption increased from 36.4 to 83.1% as the adsorbent dose increased from 0.2 to 1.0 g/100 mL.</li> <li>• Overall, the adsorption of dyes by the adsorbent treated with the addition of silver nitrate was 79-96%, the adsorbent treated with the addition of sulfuric acid was 75-89%, and the adsorbent of raw cassava peel alone was 44-60%.</li> </ul>
<p>Supporting Materials:</p>		
<ul style="list-style-type: none"> <li>• Cassava Peel</li> <li>• Silver Nitrate</li> <li>• Sulfuric Acid</li> </ul>		

### 3 Conclusion

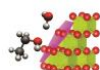
Based on the results of a literature study, cassava peel has the potential and attractiveness to be used for biocoagulant fabrication. The characteristics of the cassava peel-based coagulant greatly affect the performance in wastewater treatment. However, in general, coagulant based on cassava peel in single coagulant form or in the form of dual/modified coagulant of cassava peel has potential as an effective bio-coagulant in the treatment of removing pollutants (dyes, harmful ions) from wastewater. Because the raw material comes from waste, its application in the processing process is expected to be commercially feasible.

### Acknowledgement

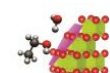
This study acknowledged RISTEK BRIN for Grant-in-aid Penelitian Terapan Unggulan Perguruan Tinggi (PTUPT) and Bangdos Universitas Pendidikan Indonesia.

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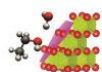
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