



Comparative study of the use of starch from agro-industrial materials in the coagulation-flocculation process

Estudio comparativo del uso de almidón a partir de materiales residuales agroindustriales en el proceso coagulación-floculación

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Abstract

Coagulation-flocculation is a process that takes place in the water treatment industries with the aim of reducing the repulsive potential of the double electric layer of colloids using the addition of coagulants. This study evaluated the use of cassava (*Manihot esculenta*), yam (*Dioscorea alata*) and plantain (*Musa paradisiaca*) starch, post-harvest residual, in the coagulation-flocculation process for the removal of turbidity in a synthetic water sample. The extraction of the starches was carried out by two methods: alkaline with NaOH and with deionized water. The starches obtained were characterized by physical analysis (color, state and pH), quantifying the nitrogen and carbon content. The effect of the coagulant concentration and the stirring rate on the coagulation-flocculation capacity of the starches under study were assessed as well. It was found that the plantain starch, obtained by both methods, reached the best percentages of turbidity reduction up to 94.6%, becoming an alternative to synthetic coagulants. The results presented in this basic study can be of great value for the scaling of starch production from these residues at an industrial level in the department of Bolívar, Colombia.

Keywords: bio flocculant, turbidity, polysaccharide, colloid, water treatment.

Resumen

La Coagulación-floculación es un proceso que se lleva a cabo en las industrias de tratamiento de agua con el objetivo de reducir el potencial repulsivo de la doble capa eléctrica de coloides usando la adición de coagulantes. En esta investigación se evaluó uso de almidón de yuca (*Manihot esculenta*), ñame (*Dioscorea alata*) y plátano (*Musa paradisiaca*) residuales postcosecha en el proceso de coagulación-floculación para la remoción de turbidez en una muestra de agua sintética. La extracción de los almidones se realizó por dos métodos: alcalino con NaOH y con agua desionizada. Los almidones obtenidos se caracterizaron por análisis físico (color, estado y pH), cuantificando el contenido de nitrógeno y carbono. Se evaluó el efecto de la concentración de coagulante y la velocidad de agitación sobre la capacidad de coagulación-floculación de los almidones en estudio; encontrándose que el almidón de plátano, obtenido por ambos métodos, alcanzó los mejores porcentajes de reducción de turbidez hasta en un 94,6%, convirtiéndose en una alternativa a los coagulantes sintéticos. Los resultados presentados en este estudio básico pueden ser de gran valor para el escalamiento de la producción de almidón a partir de estos residuos a nivel industrial en el departamento de Bolívar, Colombia.

Palabras clave: biofloculante, turbidez, polisacárido, coloide, tratamiento de aguas.

1 Introduction

Starches are important plant polysaccharides, which are a source of high availability and low cost. They also have interesting physicochemical properties such as biodegradability, biocompatibility and non-

toxicity (Almario, Mendoza-Fandiño, & Arrieta-Torres, 2019; Yuliana, Huynh, Ho, Truong, & Ju, 2012). Accordingly, applications have been found in various industries such as pharmaceutical, biomedical and polymer (Almario *et al.*, 2019; Rodríguez-Soto, Piñeros-Castro, & Ortega-Toro, 2019).

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Recently, different studies have found that due to their properties, starches can be used as flocculants, which are used as additives in a large number of industrial applications, including water treatment, food and beverages, mining, fermentation, dyes and textiles (Salehizadeh *et al.*, 2018).

The demographic growth and consequent consumption of water in different activities have increased the amount of residual effluents and pollution levels; wastewater is collected in sewage systems and purified by physical, biological and chemical processes (Pantoja-Espinosa *et al.*, 2015). The chemical coagulation-flocculation process consists in the removal of suspended particles in water with an average size of 5 to 200 nm (colloidal particles), and depends on factors such as temperature, ionic strength, pH, type and dose of coagulant material, size and type of distribution, concentration and properties of organic materials and colloidal particles in suspension (Sohrabi *et al.*, 2018) According to this, the flocculants act by attracting other colloids and other particles, in a liquid phase, to each other to form larger particles (flocules) promoting the separation of these particles from the stable suspension (Salehizadeh *et al.*, 2018). Therefore, they have been applied extensively for the removal of turbidity, dissolved and suspended solids, decrease in chemical oxygen demand in clarification and sedimentation processes (Teh *et al.*, 2016) (Lee *et al.*, 2014).

Traditionally in the chemical conditioning of wastewater, polymeric flocculants and inorganic coagulants have been used; those flocculating agents are expensive, and generally consist of metal salts of synthetic origin, which cause affectations in the bodies of water if they are disposed without previous treatment, since their stability to the cut and biodegradability are low (Kamar *et al.*, 2015); also, they affect human health since their active substances are carcinogenic, they cause poisoning and neurotoxicity (Patale & Pandya, 2012). Examples of these are ferric chloride (FeCl_3), aluminum hydroxy chloride (PAC), aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$), among others (Guo & Chen, 2017)

Despite its effective performance, it has been possible to observe the entrainment of large quantities of inorganic ions, which decreases the useful life-time of wastewater treatment equipment due to corrosion, fouling and clogging (Gao *et al.*, 2009). In addition, the widespread use of $\text{Al}_2(\text{SO}_4)_3$ and PAC has been found as a potential culprit in the development of Alzheimer's disease (Campbell,

2002). In addition, low molecular weight Al species can even penetrate biological membranes, threatening any living organism (Yusoff *et al.*, 2018). Thus, there has been a growing interest in developing flocculants that are biodegradable and based on natural products.

Starch is one of the most abundant natural polymers in the world, and its use as a coagulant has been investigated due to its applicability, abundance, renewability, low cost and biodegradability of starch, and in its raw form, it consists of a mixture of two polymers of anhydroglucose, amylose and amylopectin units, so it is an interesting material to be used as a coagulant due to its electrolytic nature (Oladoja, 2015; Teh *et al.*, 2016). The performance of the final application of starch as a coagulant-flocculant, depends largely on the structure and the molecular weight, in addition to environmental parameters (Wu *et al.*, 2016). Therefore, it is significantly important to effectively build and exploit the structure-activity relationships which influence the selection and design of high-performance flocculants in decontamination, because colloids in the water have a negative charge and a cationic flocculant is suitable for load neutralization and favourable for flocculation (Oladoja, 2015; Wang *et al.*, 2013). However, little has been studied about the effects of structures of polymeric flocculants that impact process performance due to the high size of chains (Wu *et al.*, 2016). Thus, several natural polyelectrolytes have been exploited as coagulants or flocculants in the treatment of water and wastewater, such as rice bagasse (Guo & Chen, 2017), *Tamarindus indica* (Mishra & Bajpai, 2006), *Moringa oleifera* (Rodiño-Arguello *et al.*, 2015), *Manihot esculenta* (dos Santos *et al.*, 2018), *Malva sylvestris* (Anastasakis *et al.*, 2009), *Cactus opuntia* (Fuentes-Molina *et al.*, 2016), guar gum, tannins, sodium alginate and pectin are among the natural sources that have been examined, starch being one of the most studied (Oladoja, 2015).

It has been reported that the components of different species of plantain, cassava and yam, in order from highest to lowest, are: starch, moisture, fiber, lipids, proteins and ashes (Chukwudi *et al.*, 2008; Salcedo-Mendoza *et al.*, 2016; Trujillo *et al.*, 2014). Thus, based on the characterizations reported in the literature of yam, banana and cassava starches (FTIR, SEM, XRD, chemical and elemental analysis), these exhibit double helix arrangements produced by the semi crystalline distribution of starches, which influences the formation of the flocs and their subsequent sedimentation (Ashri *et al.*, 2014; Chávez-Salazar, Bello-Pérez *et al.*, 2017) Therefore, it is stated

that the species (*Manihot esculenta*), yam (*Dioscorea alata*) and plantain (*Musa paradisiaca*), native to Colombia, they can also be good natural coagulants.

The current investigation developed the comparative study of the coagulation power of starches coming from three natural raw materials: cassava (*Manihot esculenta*), yam (*Dioscorea alata*) and plantain (*Musa paradisiaca*). The starches were obtained from post-harvest residues of the Colombian Caribbean coast, for which the supplied raw material met the condition of not being able to be sold or used as food, mainly due to its poor postharvest management, not allowing the product to have a good quality.

2 Materials and methods

2.1 Materials and equipment

During the experimental procedure, Merck-brand analytical grade reagents were used: sodium hydroxide, ferric chloride, distilled water, analytical balance, Lovibond PFX195 digital colorimeter, jar test equipment, centrifuge, Bante 900P multi-parameter pH meter, Thomson WGZ 400B turbidimeter.

2.2 Design of experiments

In this research, an experimental response surface design of the central star composite type was developed, with the aim of verifying the coagulating power of the starches of the former mentioned raw materials. Two extraction procedures were used for these starches, in order to compare their effectiveness as well. One through an alkaline solution of sodium hydroxide and another with distilled water.

The response variables established for this purpose were pH, color and turbidity of the water samples studied. There were also established as independent variables the stirring rate in two levels (30 and 40 rpm) and the concentration of coagulant in three levels (125, 187.5 and 250 ppm). The intervening variables that were kept constant through experimentation were: the initial concentration of the sample, the centrifugation time (10 min), the centrifugation speed (1500 rpm) and the cooling time (20 h).

2.3 Starches extraction

Firstly, 1000 g of each of the residual post-harvest materials (cassava, yam and plantain) were taken

and processed for further washing with distilled water in order to eliminate impurities. Then, 500g were immersed in a solution of 0.25% (p/v) NaOH and 500g in water (in order to compare both, the alkaline extraction and that of the process by wet hydrolysis). Both mixtures were cooled at 4 °C for 20 h, and subsequently liquefied for 1 minute. After the maceration process, products rich in starch were obtained, which were filtered separately.

The filtrates were centrifuged at 1500 rpm for 10 min. The centrifuged material was adjusted to neutral pH using a 2M HCl solution and centrifuged again under the same conditions. Finally, the starch was dried for subsequent size reduction by milling (Maniglia & Tapia-Blácido, 2016).

2.4 Starch characterization

After extraction, the starches were characterized by determining the nitrogen and carbon content by means of the AOAC 949.12 and AOAC 984.13 methods, respectively. Similarly, color and physical state analyses were performed, and pH was determined using a 900 Bante Instrument Multiparameter.

2.5 Synthetic water preparation

For the obtention of synthetic cloudy water, 0.3 g of bentonite were added in two containers with 800 ml of deionized water. This solution was mixed for 1 h at 200 rpm, and subsequently left at rest for a hydration process. Then, they were diluted in 1.9 L each and mixed resulting in a cloudy water of 32.3 NTU (Canepa, L; Maldonado, V; Barrenechea, A; Aurazo, 2004).

2.6 Assessment of the coagulant capacity of starch

The turbidity and color removal tests were carried out by placing the turbid water sample, which had an initial pH of 6.8, in contact with the starch extracted and aluminum sulfate in the equipment for jar test at 200 rpm for 1-2 min and, at the different coagulant concentrations (125, 187.5 and 250 ppm) established in the experimental design; then the stirring rate was reduced to 30-40 rpm for 15 min, the mixture was allowed to stand. Then, an aliquot was taken to measure the turbidity (NTU), the color (Pt/Co) and the pH of the solution.

2.7 Statistical analysis

The effect of pH, agitation speed and the initial dose of starch on coagulation capacity was analysed using Minitab 17 Software.

3 Results and discussion

3.1 Extraction and characterization of starch

Applying the process of extraction of starches detailed in the methodology for cassava, yam and plantain by hydrolysis, the results of Table 1 were obtained.

The investigations of starch extraction present a yield of extraction around 0.5% (Carrasquero *et al.*, 2017), and 1.5% (Hernández *et al.*, 2017) for the hydrolysis process. Therefore, the percentages

of starch extracted are optimal since they exceed the percentages reported, with yam starch being the highest percentage with 16.26%.

Table 2 shows that the starches have a high percentage of carbon, which is related to the efficiency of coagulation. From the data obtained from the carbon and nitrogen parameters, it can be noted that all of the varieties have similar percentages of carbon, with cassava starch having the highest percentage using both methods of extraction, with a minimum difference. Thus, the 3 species they would present a similar efficiency as coagulant.

Table 1. Weight of starches according to the aqueous and alkaline treatments.

Biomass	Aqueous method		Alkaline method	
	Weight (g)	Yield (%)	Weight (g)	Yield (%)
Cassava	68.1	13.62	35.5	7.1
Yam	81.3	16.26	78.2	15.64
Plantain	35.5	7.1	38	7.6

Table 2. Characterization of the cassava, yam and plantain starches obtained by the aqueous and alkaline treatments.

Method	Biomass	Carbon (%)	Nitrogen (%)	Physical state	Color	pH
Aqueous	Cassava	47.08	0.18	Solid	White Ivory	7
	Yam	46.92	0.28	Solid	White Ivory	7
	Plantain	46.98	0.24	Solid	Light Brown	7
Alkaline	Cassava	47.68	0.11	Solid	White Ivory	7
	Yam	47.34	0.18	Solid	White Ivory	7
	Plantain	46.9	0.15	Solid	Dark Brown	7

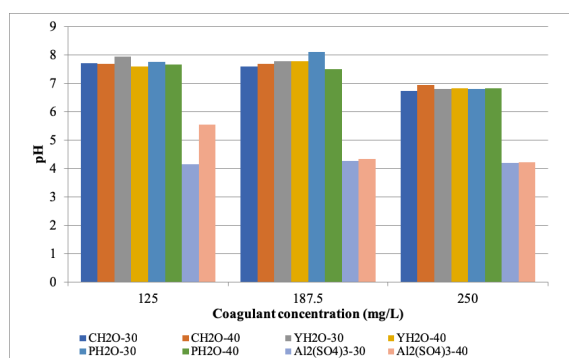
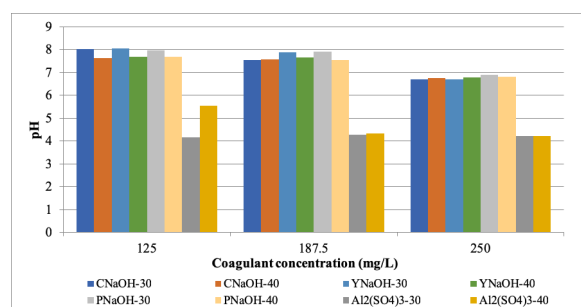


Fig. 1. pH behavior varying the coagulant concentration and stirring rate. (a) Alkaline extraction (b) Wet extraction.

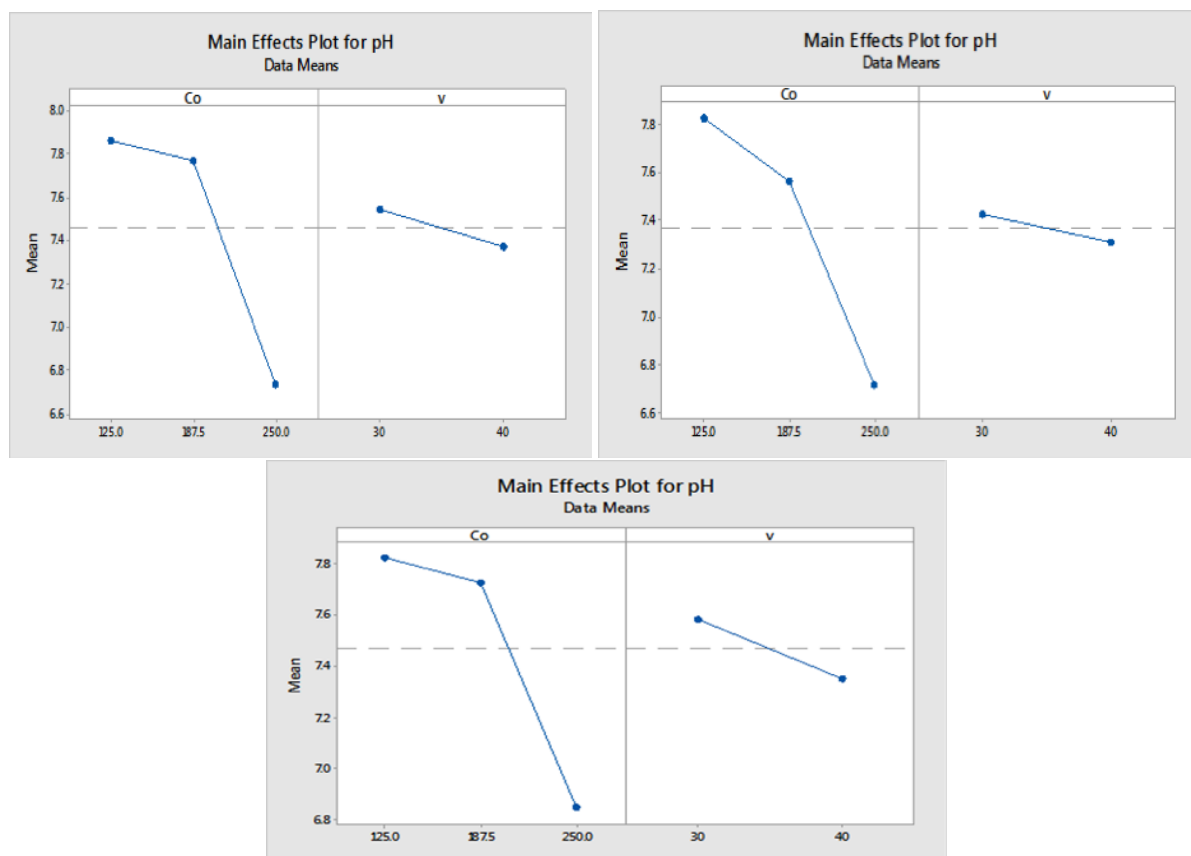


Fig. 2. Graphs of main effects for the pH of the turbid water regarding the concentration of coagulant and stirring rate. (a) cassava starch, (b) yam starch, (c) plantain starch.

3.2 Effect of the type of starch on the pH of the water

Figure 1 shows the comparison of the pH of the raw water in the tests with the starches coming from the three raw materials, both for the alkaline extraction and for the one made by hydrolysis, at different concentrations of coagulant and stirring rates. The nomenclature of the plotted data was defined as the first letter for the raw material (C: Cassava, Y: Yam, P: Plantain), followed by the extraction method (NaOH: alkaline, H₂O: humid) and finally the stirring rate used in the jar test (30 and 40 rpm).

It can be seen in Figure 2, from the graphs of main effects generated by means of the software Minitab 17, that the pH is not affected clearly by the stirring rate. Also, it is inversely proportional to the concentration of coagulant.

Similar behavior was detected in both alkaline and wet extraction methods. There is also a similarity in the behavior of the pH according to the type

of raw material of each starch, with cassava starch generating, on average, slightly more basic pH values. This behavior could be due to the fact that with an increase in pH, the zeta potential of the starches is more negatively charged by the presence of the carboxyl groups of the proteins (COO⁻) (Choy *et al.*, 2016).

The pH is an important parameter in the coagulation process, since it can change the surface charge of the coagulant and/or contaminant (Kukić *et al.*, 2015). In addition, pH dependence has also been reported in the literature for horse chestnut and acorn as natural coagulants (Šćiban *et al.*, 2009) but not with bean seed extracts (Kukić *et al.*, 2015), and plantain peel powder (Daverey, Tiwari & Dutta, 2018). This implies that the natural coagulants obtained in the present study are highly stable over a wide pH range and can be used as coagulants for the treatment of real samples of water and wastewater (Paredes *et al.*, 2018).

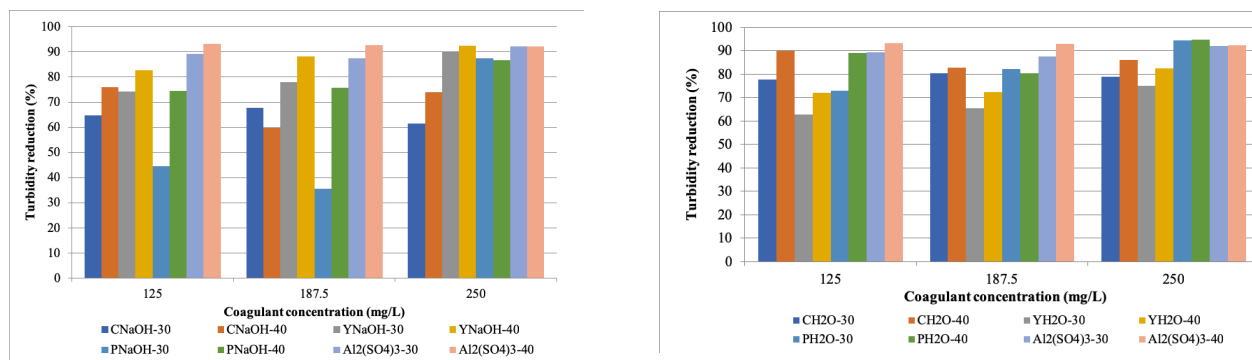


Fig. 3. Percentage of turbidity reduction regarding the concentration of coagulant and stirring rate. (a) Alkaline extraction (b) Aqueous extraction.

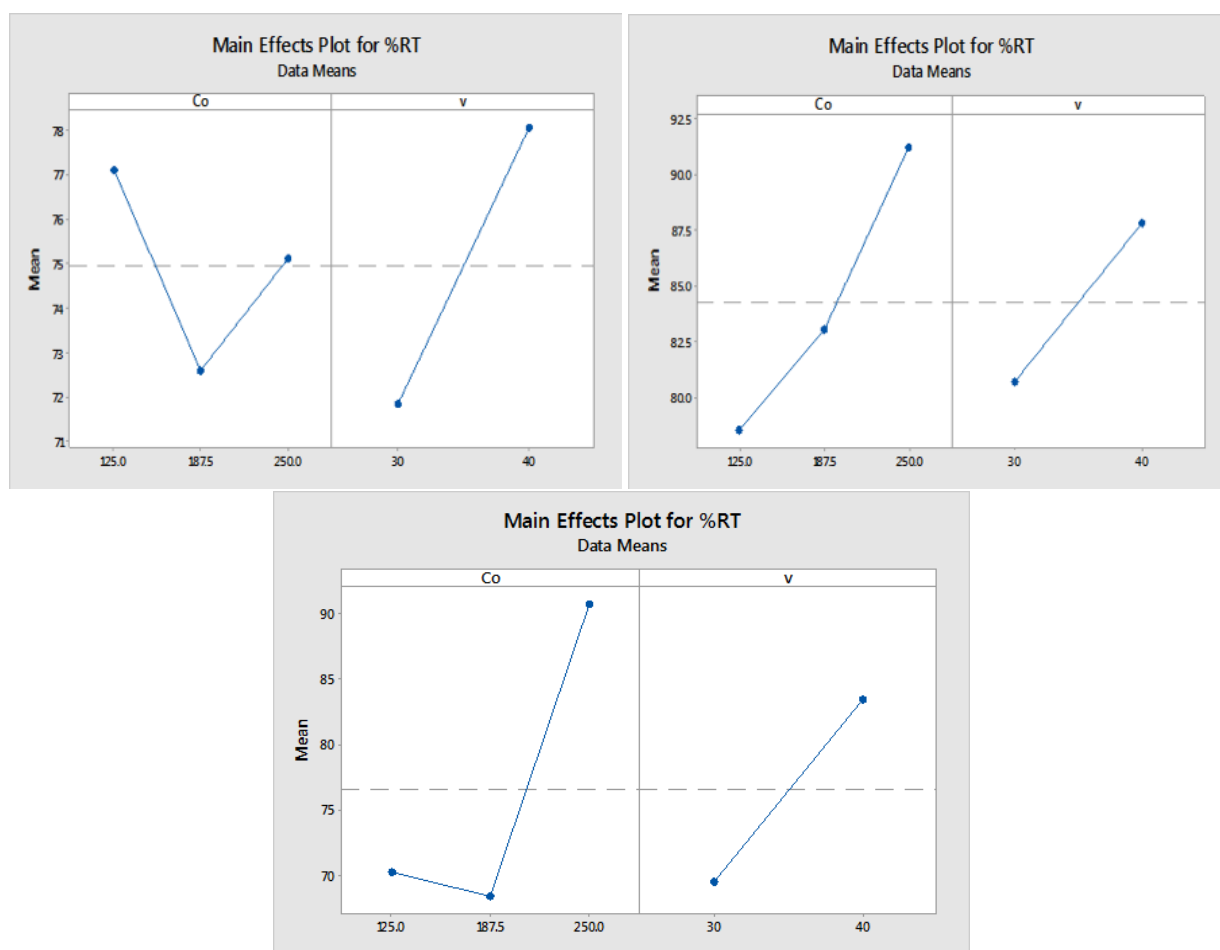


Fig. 4. Graphs of main effects for the reduction of turbidity regarding the concentration of coagulant and stirring rate. (a) cassava starch, (b) yam starch, (c) plantain starch.

3.3 Effect of the starches on the turbidity reduction

Figure 3 shows the percentages of turbidity reduction evaluated at three different concentrations of coagulant and stirring rates for each type of starch and aluminum sulfate. This percentage is based on the initial value of 32.2 NTU for the turbid water prepared according to the methodology.

It is evident that, on average, the starches extracted through the wet method showed a higher level of turbidity reduction than those obtained through the alkaline method. On the other hand, while considering the type of raw material, it can be observed that the three varieties of starch reached similar turbidity reduction values.

On average the starch extracted from yam presented more favorable results in this aspect with a 77.9% reduction, followed by plantain starch with 76.5% and finally cassava with 74.9%. Amylopectin, which is a branched fraction of the proteins present in the starch, would help in coagulation, similar to those present in other natural coagulants such as Maerua subcordata (Mavura *et al.*, 2008), rice, wheat, maize and potato (Choy *et al.*, 2016).

When comparing the yield of the starches with respect to the commercial coagulant Al_2SO_4 , the plantain starch extracted by the wet route being used at the highest concentration of coagulant evaluated (250 mg / L) which reached the maximum turbidity reduction of 1.72 NTU. These ones were above of the values reported for the aluminium salt at the same conditions, this due to the rapid formation of floccules that contributed to sedimentation due to the consistency and weight of the flocculated particles, in accordance with Paredes *et al.* (2018).

In Figure 4 it can be seen that there is no clear correlation for the concentration of coagulant for cassava and plantain starches. On the other hand, there is a directly proportional relationship between concentration of coagulant and percentage of turbidity reduction for yam starch. It is also noted that for the three varieties of starch the increase of the stirring rate established a rise on the percentage of turbidity removed.

Conclusions

It can be concluded that the starch extraction process was efficient for the three types of biomass evaluated

(cassava, yam and plantain) since the extraction yields were higher than those reported in previous investigations; in turn, the yam was the species that presented the highest percentage. An inverse correlation was found between the pH and the concentration of coagulant, while the stirring rate had no appreciable effect on this response variable for any of the starches. There was no addition or degradation of color in the turbid water for any of the coagulants. Regarding the percentage of turbidity reduction, it was found to be proportional to the stirring rate, with the best performance ratio being plantain > yam > cassava, presenting plantain starch better behavior as a natural coagulant than that of yam and yucca, and even commercial coagulant evaluated with 94.6% efficiency.

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Nomenclature

C	Cassava starch
Y	Yam starch
P	Plantain starch
rpm	Revolutions per minute
NTU	Nephelometric Turbidity Unit

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