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Using Eco-Friendly Nano-Polymers in Industrial Wastewater Treatment

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Abstract. The major target of this research is to synthesize an eco-friendly coagulant, based on a biomaterial (potato starch) that contribute in industrial wastewater treatment. From this work an eco-friendly nano-polymer was produced from potato starch. Successfully not only the potatoes were used, but also the potato peel, which has an economic impact on reducing the amount of water wasted in planting such an important crop as the potatoes all over the world. Because of increasing the need for food production and changing the dietary pattern, the development of potato industry has become the first among all food crops in most of the countries. As a result potato starch became the most popular type among the polysaccharides. To achieve this, the experimental work started by extracting the starch from the potato and potato waste (peels). In order to enhance the potato starch characteristics, the synthesis of starch nano-particles (StNPs) was conducted by gelatinization using a mixture of sodium hydroxide along with glycerol in aqueous medium throughout the reaction process, while settling down the StNPs which was done by homogenization at ambient temperature. Characterization was done on both native potato starch and starch nano-particles using Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and Energy Dispersive X-ray Spectroscopy (EDX). The second part of the experimental work is to test the eco-friendly starch nano-particles (starch-based coagulants) in industrial wastewater treatment, and to evaluate its effectiveness as a coagulant against the most commonly used synthetic polymer (PolyDADMAC) playing the same roll in industry and whether it can really replace it or not.

Keywords: Polysaccharides, Starch nanoparticles, Starch-based coagulants, Industrial wastewater treatment, PolyDADMAC

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1. Introduction

Coagulation has a major role in wastewater treatment innovation. It is one of the most commonly used productive and economical techniques in treating the wastewater, as well as for sludge dewatering [1]. Colloidal particles are considered a major concern among all other impurities, this is due to its electrical charge carried on its surface and because of the very small size leading to repetitive collisions with the dispersing medium; that will keep the colloidal particles suspended and not settled. Additionally, it is causing unwanted turbidity of water supply, along with preventing the light entrance cure and disrupting the aquatic ecological community [2]. In order to dispose these colloids, coagulation is being selected over the world due to its approximately low-priced, manageability and high productivity. Since protecting the environment is the most restricted prerequisite, inorganic and synthetic organic polymer coagulants are not recommended for their indissolubility and the chance of causing of contamination. Metallic coagulants including aluminium and iron-based salts, such as aluminium sulphate, poly-aluminium chloride and ferric sulphate are commonly used because of their extended approachability and great coagulation effectiveness [3]. Regardless of the fact that coagulants have a highly praised capability, the demand of these coagulants can cause releasing of the poisonous aluminium into the aquatic environment. Large mass of aluminium in treated water has made more worries about the effect on health because of long exposure to aluminium in drinking water that may eventually cause Alzheimer disease [3].

In consequence, the requirements for more eco-friendly coagulant are expanding. Other varieties of justifiable natural biopolymer coagulants have been continuously enhanced and published. Natural polysaccharides, as in: starch, chitosan, cellulose and lignin [4], are universally utilized as organic polymer coagulants due to their ability to enhance the coagulation characteristics, thus adjustments of these natural polymers are always needed. Starch, as one of the ultimate suitable and affordable polysaccharides, is an encouraging applicant for sustainable materials. The enhancement of starch is accessible with the reactive groups on the starch backbone. A lot of modification methods have been done on starch, for example: acylation, esterification, oxidization, cross-linking and grafting [4]. However, using the non-polluting chemicals and the joining between mechanical and chemical processes is more advantageous in improving the original starch properties and transforming it to nano-particles used as coagulant in industrial wastewater treatment.

2. Materials and Methods

2.1 Materials and Equipment

Distilled water, Potatoes, Ethanol (99% concentration), Glycerol, Sodium hydroxide, Tween 80, 1000ml flask, Set of sieves, electronic scale, Mechanical stirrer, Filter cloth, and Whatman 50 filter papers.

2.2 Preparation of potato starch

The experiment started by good washing of 1000g of potatoes, followed by separating the potatoes from its peel, then the weight was measured by using an electronic scale, which was about 821g of raw potatoes. Potatoes were grinded to obtain a mixture involving starch flacks and the potato juice, which it is a combination of proteins, amino acids, sugars and salts. A little amount of dripped water is added, then mixture is stirred manually for 30 minutes. Starch flacks and non-starch polysaccharides were separated from the solution by sieving, followed by using a filter cloth to apply filter press. This procedure was repeated for three times. The remaining filtered water kept for 20 min to make sure that the starch will be obtained by sedimentation. The obtained starch from this procedure, was dried and then measured again (about 49.46 g of dry starch), the percent yield of starch extracted from potatoes was 0.25%.The same procedure was done on the left potato peel. The starting weight of the peel after washing was 465.94 g while the obtained dry starch from potato peel was about 8.38 g, the percent yield of starch extracted from potato peel was 0.09%, which can make the potato peel a suitable source instead of the original potatoes.

2.3 Synthesis of starch nanoparticles (StNPs)

This procedure (El-Sheikh, M. A. (2017)) depends on gelatinization of the native starch (NS) prepared previously from potato peel, by sodium hydroxide and glycerol. Starting by dissolving 1.5 g of sodium hydroxide and 2.7 g of glycerine into 100 ml of distilled water, then continue addition of 5g of the native starch by portioning the quantity, while stirring of the mixture at the same time. After receiving a clear slurry of the consistent gelatinized starch, it is kept for stirring at 25 ◦C for fifteen minutes. Then, the entire mixture was left to settle down for fifteen minutes at 25 ◦C. Followed by slow dropping of 100 ml of pure ethanol into the mixture and eventually 10 drops of Tween80 were injected as an emulsifier as well. The gelatinized mixture of starch was stabilized by stirring for fifteen minutes. A milky colour sediment was produced and then filtered after a few time using whatman 50 filter papers. The output from the previous step was rinsed several times using 80:20 ethanol: water ratio, to remove any traces of sodium hydroxide, continued with one last rinse using pure ethanol and kept for drying at 25 ◦C.

Figure 1. Native Starch (NS)

Figure 2. Colloids of Starch nano-particles (StNPs)

2.4 Coagulation Experiments

Jar tests were done to simulate the process of coagulation and wastewater treatment at the laboratory, and to test the effectiveness of the coagulant (Aluminium sulphate) along with the coagulant-aids (Industrial starch, prepared NS, StNPs and PolyDADMAC) in synthetic wastewater treatment at different parameters until the optimum condition was reached. To study the effect of contact time, the coagulant (aluminium sulphate) was freshly prepared with constant concentration of 10% at 30ppm, and the jar test started firstly at 200 rpm for 15 min, followed with addition of coagulant-aid with constant concentration of 2.5% at 1ppm and 100 rpm, at different contact times (0-20 min). The optimum contact time was at 10 min, and StNPs achieved the highest percentage removal with 95.3%. To study the effect of coagulant-aid dosage (0-4.5 mg/l) the contact time was constant at 20 min, the optimum dosage was at 2mg/l, and StNPs was the highest in its percentage removal with 97.6%. As for the effect of pH of the synthetic raw water, it was tested from (0-14) on pH meter, and the optimum was at pH=7, with highest percentage removal of StNPs =95.1%. The percentage removal was calculated using the equation: Removal% = $\{T_o\text{-}T_e/T_o\}*100$, were T_o and T_e are the values of turbidity before and after coagulation.

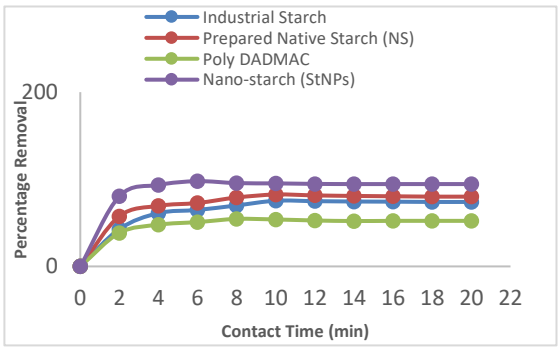

Figure 3. Effect of contact time of coagulant aid

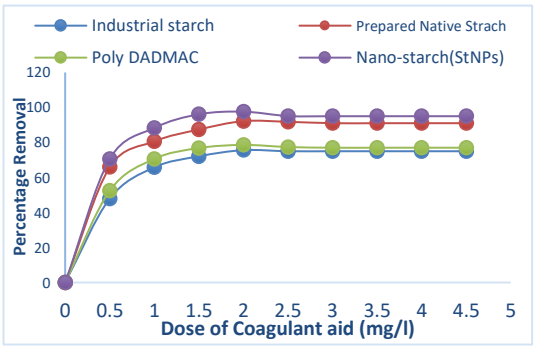

Figure 4. Effect of coagulant aid dosage

2.5 Methods

2.5.1. Scanning Electron Microscope (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX): This test was done using (JSM-IT200 Series). It is a sort of electron microscope that creates pictures of a specimen by examining the exterior with an immersed ray of electrons. The electrons interfere with atoms in the specimen, creating different signals expressing data about the surface topography and composition of the specimen. As the EDX is attached to the same testing tool, it is helping to analyse elemental or chemical composition of the specimen, Its performing potentialities are because of the huge principal standards where every single element has a particular atomic formation releasing a particular batch of peaks on its electromagnetic transmission spectrum[5].

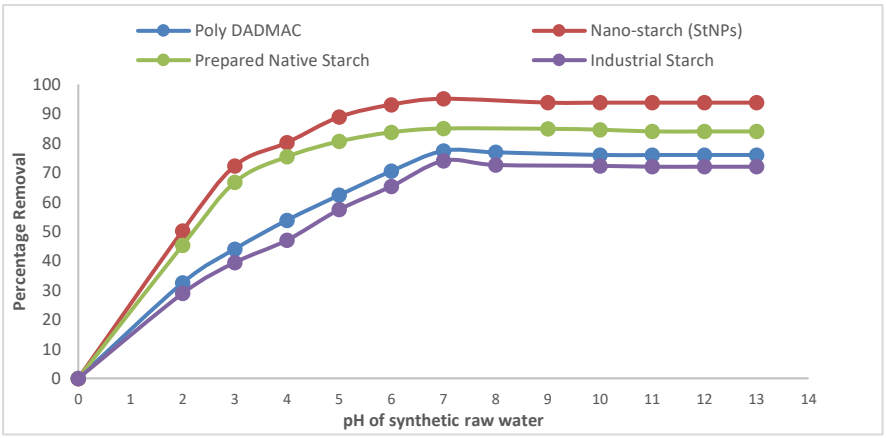

Figure 5. Effect of pH of synthetic wastewater

2.5.2. Fourier Transform Infrared Spectrum (FTIR): This test was carried out using (Cary 630) FTIR. By using this method, the molecular structure can be detected to target the difference between native and nano-particles of starch. Along with determining how the cells of the starch are affected during the synthesis of nano-particles, specifying the destructed cells from the unaffected ones [6].

2.5.3. X-Ray Diffraction (XRD): It`s a quick scientific method using (Bruker AXS D2 PHASER) initially applied to determine the degree of crystallinity of both NS and StNPs and also offer data about the unit cell measurements. The examined matter is stabilized, and intermediate bulk content is calculated [7].

3. Results and Discussion

3.1 Fourier Transform Infrared (FTIR) Spectroscopy

The evaluations show that featured apexes in the FTIR spectra can be observed for every single sort of potato starch. It is extremely helpful to investigate manufactured starches which are enhanced by human means. From figure 6 it appears that a wide band in the IR spectra of NS with two highest points at 3243.1 and 2927.6 cm^{-1} were observed, which represent the stretching mode of the O-H found in the dried starch. The major absorption bands 1636.2 – 706.3 cm^{-1}. The surge 1418.8 cm^{-1} is linked to the C-H bending of CH_2 and the surges at 1244.6 and 1339.7 cm^{-1} are related to O-H bending of primary or secondary alcohols. As observed from figure 6 the NS extracted from the potato peel has almost the same IR spectra with only slight decrease or increase, except for the severity of the broad O-H band at 3645.1 – 3000 cm^{-1} is extremer than starch extracted from the original potato, which is offering an elevated density of strong hydrogen bonding interactions in this kind of starch [8]. By comparing the results to researches done, the final result was surges appearing at wavenumbers located between 3100 and 3700 cm^{-1} shows the O-H bonds (the alcohol bonds), which are most of the time observed in both native and hydrolysed potato starch. Correspondingly, the wavenumber located between 2920 and 2928

cm^{-1} is proof of availability of CH_2 bonds (methylene linkages) in the starch molecules, which are not present in native potato starch but available in hydrolysed potato starch [9].

The StNPs extracted from potato peel, were characterized using FTIR in figure 6, showing that the peaks at 760.1 cm^{-1} related to C-C group, 935.1 cm^{-1} related to α-1, 4 glycosidic groups, and 1079.1 cm^{-1} related to the C–O-H groups which are unique for polysaccharides. Moreover, the points at 1361, 1651.9, 2931, and 3122.2 cm^{-1} related to the C-H twisting mode of CH_2, bond twisting vibration stretching vibration of CH_2 [10].Comparing the FTIR results of native starch (NS) with the starch nano-particles in figure 6, the end result can be easily recognized that the key specific bands of NS and that of StNPs are nearly the same in the place of the surges with little growth/reduction in the severity of the surges of StNPs reflected to NS. Which indicate that no actual difference in the chemical structure of the starch molecule that is replaced for StNPs. It is noted that the point 3243.1 cm^{-1} for NS (O-H), where it is moved to 3453.8 cm^{-1} for StNPs. Compared with NS, the severity of the O-H band of StNPs is further down. This is caused by several reasons, as in the prevention of the hydroxyl groups as an output to the generation of sodium starchate. Another reason might be because of the waste of the crystalline structure and being affected by the O-H groups of the starch molecule [11].

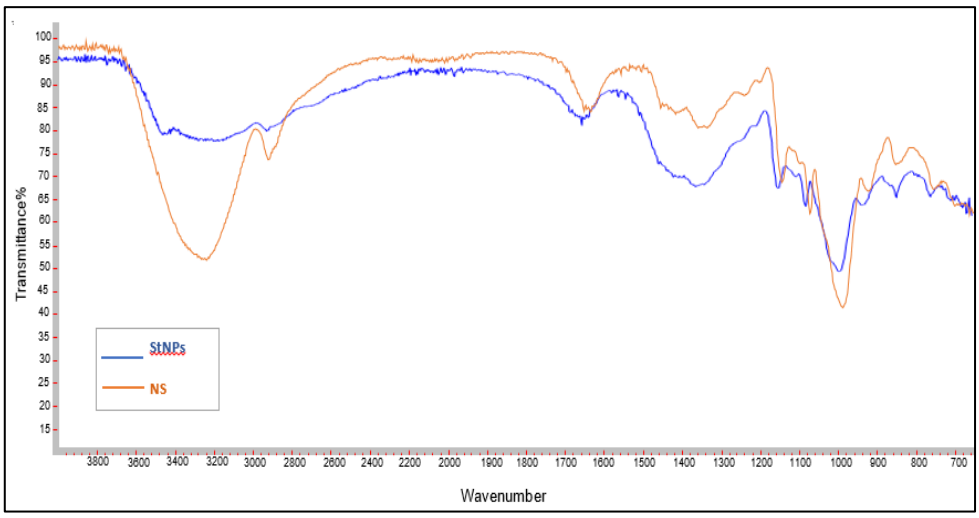

Figure 6. Infrared spectra of native starch and nano-starch from potato peel

3.2 Scanning Electron Microscope (SEM)

From figure 7 it is clearly showing the slicked surface and circular shape of the native starch particles, with sizes ranging from 12 – 52 micro-meters. It is important to note that the sizes of individual granules of potato starch is not the same according to the potato origin, and therefore SEM help to produce a detailed method of to identify starches from different potato origins [12]. Mechanical action was done on native potato starch pulled out from the potato itself, and it`s peel, resulting of nano-starch particles in figure 8. The mixture formed after processing is about 97% nano-particles with average size 15-40 nm. For presenting the form and the properties of nano-starch, ultra sonication was useful for high resolution SEM images in figure 8. The nano-particles were identified as amylopectin-type short branched type. The starch nano-particles are more sensitive to chemical reagents and have different properties than native starch granules in figure 7, so they can be used as a perfect holder in the chemical discharge systems [13].

3.3 Energy Dispersive Analysis of X-rays (EDX)

From figure 9 and table 1, it is shown that the main elemental components using EDX analyses, in potato peel starch are carbon with 50.98 mass% and oxygen with 49.02 mass%. By comparing these results to previous studies, it is shown that native starch whether it is from potato or potato peel is having the same elemental composition [14]. After subjecting the native starch for treatment, to enhance its performance and characteristics. From figure 10 and table 2, it is shown that the main elemental components in nano-starch particles are carbon about 38.9 mass% and higher oxygen about 57 mass% than the native starch, and with a minor sodium and calcium mass percent respectively about 3.77% and 0.2%[15].

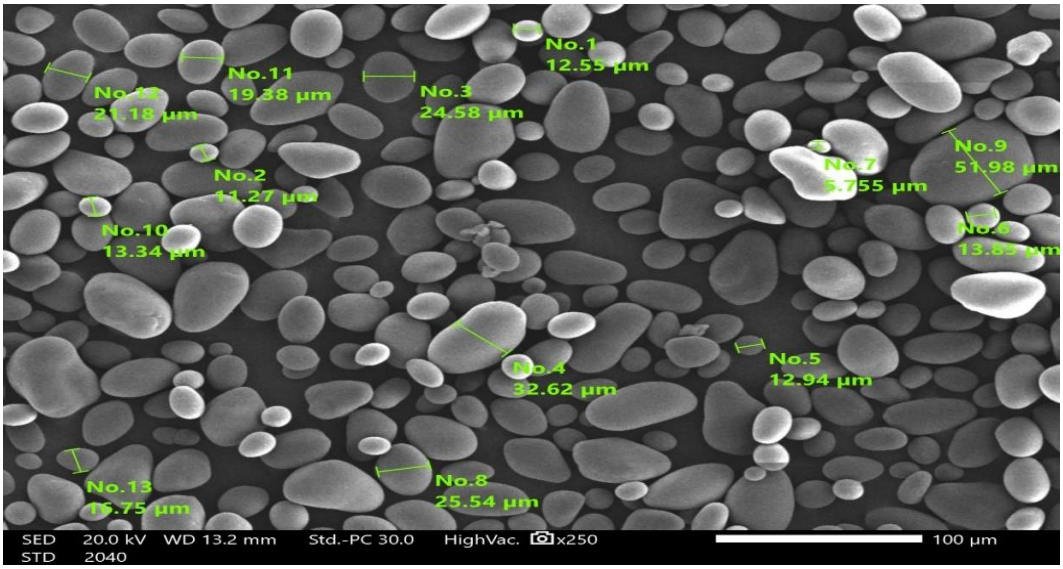

Figure 7. SEM of native starch from potato peel

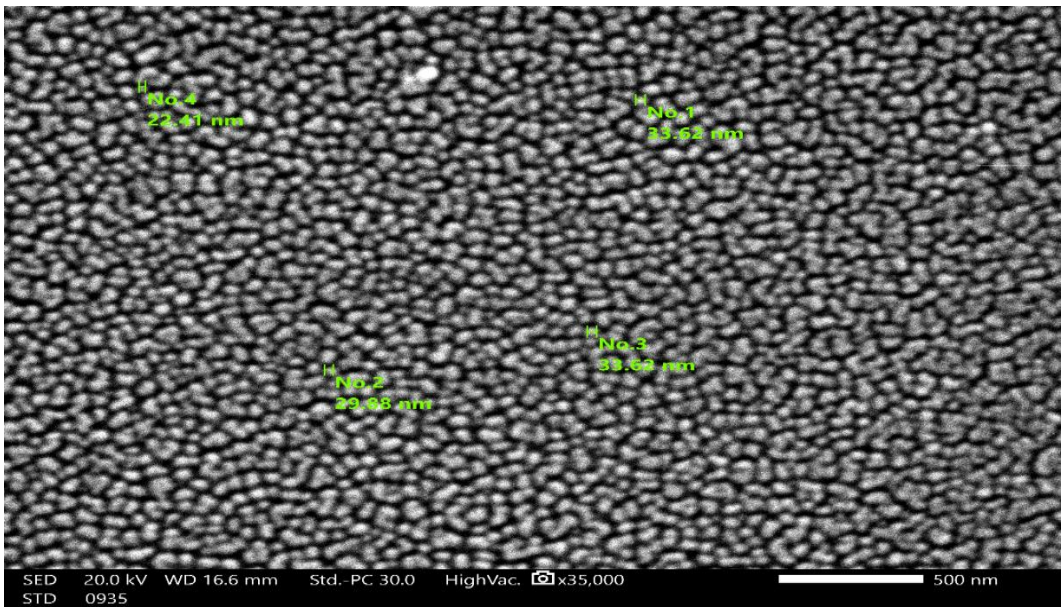

Figure 8. SEM of nano-starch from potato peel

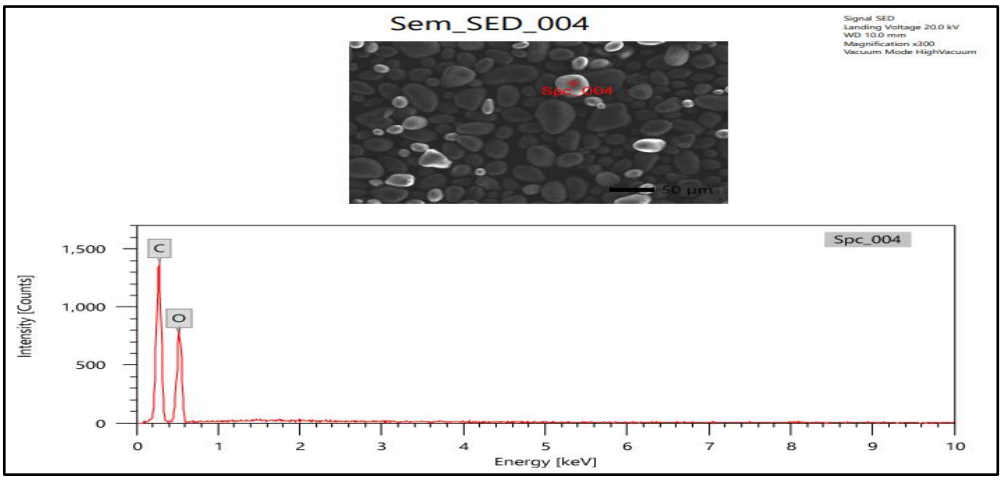

Figure 9. EDX of native starch from potato peel

Table 1. EDX of native starch from potato peel

Element	Line	Mass%	Atom%
C	K	50.98	58.08
O	K	49.02	41.92
Total		100	100

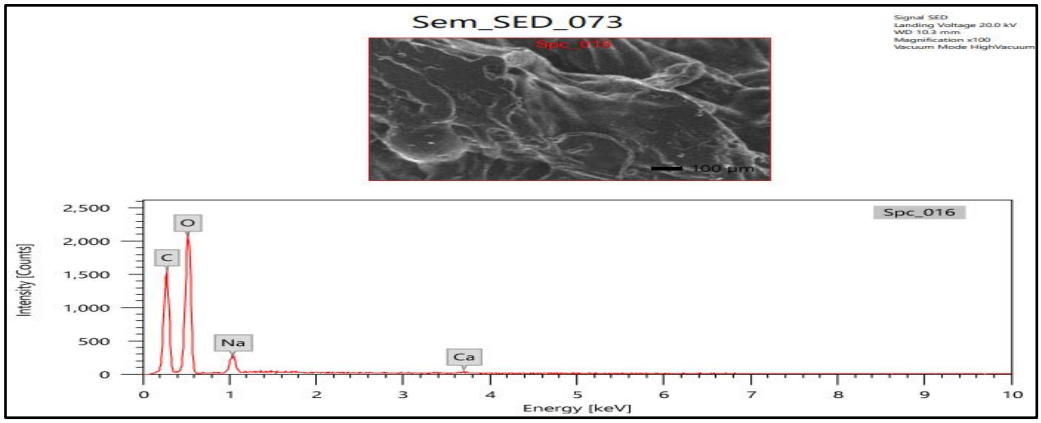

Figure 10. EDX of nano-starch from potato peel

Table 2. EDX of nano-starch from potato peel

Element	Line	Mass%	Atom%
C	K	38.86	46.38
O	K	57.14	51.19
Na	K	3.77	2.35
Ca	K	0.22	0.08
Total		100	100

4. Conclusions

The growing importance of nano-materials from natural source and their unique properties have guided this research to focus on nano-sized particles from natural polysaccharide polymers, as example starch. Because of the nano-metric size effect, nano-scale coagulants similarly have higher specific surface area, causing more of self-interaction, which allow mechanical improvement at lower coagulant amount than with previous old coagulants. Industrial water treatment is such an important sector, and adding this eco-friendly and sustainable starch nano-particles will make a huge development in the future based on the scope of the study conducted in this research. For the starch nano-particles gained from the waste of potatoes and its peel, the percent yield 0.09% of starch extraction expressing that potato peel can be a perfect sustainable source, this type of action can convert potato waste into precious products. These nano-particles can be further enhanced as wanted. Coagulation experiments proved that StNPs achieved the highest percent removal among other coagulant-aid as mentioned, at the optimum conditions.

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