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CHARACTERIZATION OF WATER MELON SEED USED AS WATER TREATMENT COAGULANT

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ABSTRACT

The residue of crushed watermelon seed was characterized using Fourier Transform Infrared (FTIR) spectroscope, X-ray Fluorescence (XRF), and Scanning Electron Microscopy (SEM) to determine the functional groups present, elements present that gave it the coagulant properties, and to view the surface morphology respectively. The FTIR spectra vibrational frequencies of the crushed watermelon seed reveals the functional groups that made up the watermelon seed at the following spectra 3447, 1845, 1740, 1647, 1559, 1541, & 1419 and revealed the presence of coagulant proteins in the crushed watermelon seed. The XRF analysis of the crushed watermelon seed revealed the element present in it that gave it the coagulant properties which are Al_2O_3(20.309wt%), Fe_2O_3 (6.556 wt%), SiO_2(12.440wt%), and Na_2O (3.630 wt%). The SEM shows the backscattered detector (BSD) images of the crushed watermelon seed which shows that the specimen is in particle structures as it was prepared as finely grounded powder. It also reveals that the surface of the particle is rough, and that the white coloured spots on the image indicates regions with strong intensity and the other grey colours indicates light intensity.

Keywords: (watermelon, characterization, coagulant, residue, functional groups)

1.0 INTRODUCTION

Coagulation is one of the most vital operations in water treatment used indestabilizing suspended particles and to react with organic materials in raw water. The formation of insoluble compounds between organic matter and coagulants, as well as the adsorption on freshly formed hydroxide precipitate, could be the determining method of organic matter removal by coagulation(Yang, et al., 1999). Mineral additives containing metal salts,polyaluminium chloride, aluminium sulphate, ferric chloride and synthetic polymers such as polyacrylamide are the coagulants that are commonly used to remove organic contamination .Coagulants such as alum are commonly employed and it reduces the repulsive force between particulate matter, encouraging particle collision and floccule formation . These synthetic coagulants have a negative impact on the environment and health, examples, aluminium ion concentrations above 50µg/L are potentially toxic to fish and aquatic organisms, and polyacrylamide residues (acrylamide) are neurotoxic and poses strong carcinogenic properties whichare peripheral nerve toxins that affect man and animals (Ghebremichael, et al., 2005; Thomas & Jurgen, 2002). As a result of the negative effects of these synthetic coagulants, interest in the use of natural coagulants has increased because of their abundance, low price, nontoxicity and biodegradability.

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Natural coagulants from plant extracts such asMoringaoleifera seeds (Beltran-Heredia, et al., 2009), Cactus latifaria and Prosopisjuliflora are natural coagulants used in water treatment for human consumption(Zhang, et al., 2006). It is verified that active coagulant agents in plant extracts are proteins because of their ability to bind with other molecules tightly(Talbot, et al., 1995). Although, recent study confirms that watermelon (citrulluslanatus) seeds is a potential natural coagulant for water treatment because of its high protein content(Muhammad, et al., 2015).

Watermelon (Citrulluslanatus) is a member of the cucurbit family (Cucurbitaceae). The crop is grown commercially in areas with long frost-free warm periods and the crop may be established in the field by planting seeds or using containerized transplants. Watermelons have four distinct parts namely the rind/peel, the seed, the fleshy white and the fleshy red/pink/yellow parts. The seeds can be brown, white green, or yellow and a few varieties are actually seedless. Watermelon seeds are a source of protein, B vitamins, minerals (such as magnesium, potassium, phosphorous, sodium, iron, zinc, manganese and copper) and fat among others.

It is a well-known fact that synthetic coagulants such as aluminium-based coagulants have shown negative effect on humans such as the development of Alzheimer's disease resulting from high amount of aluminium remaining in treated water, monomers of some synthetic organic polymers are neurotoxic and poses strong carcinogenic properties which are peripheral nerve toxins that affect humans and animals. Synthetic coagulants are known to produce large amount of sludge and altering the chemistry of the treated water, thereby changing its pH. Hence, thisstudy is aimed at the characterization of watermelon seed as a potential coagulant for water treatment.

Recent studies have indicated a number of serious shortcomings linked to the use of synthetic coagulants such as development of Alzheimer's disease, excessive sludge production during water treatment and considerable changes in water chemistry due to reactions with the OH– and alkalinity of water. Synthetic coagulants are seldom used in some developing countries due to high costs of importation and low availability. A number of studies have indicated that the introduction of natural coagulants (plant-based) as a substitute for metal salts may ease the problems associated with chemical coagulants because it is readily available, it produces less sludge, and the cost of water production using it is low. It therefore becomes necessary to search for other plant materials exhibiting these coagulant properties for water treatment in order to make it safe for humans.

Historically, natural coagulants made from vegetables were used in water treatment for drinking before the introduction of chemical synthetic base aluminium and iron. There is indication of the use of nirmali (S. potatorum) and nuts from 400 AD in India. For this purpose, in the sixteenth century, almonds soaked beans was used as coagulant in Egypt and Sudan to improve water quality .

2.0 MATERIALS AND METHODS

2.1 MATERIALS

In this work, the following were materials used. They included Bowl, Distilled water, Drying oven(GENLAB model: P811C), Electronic weighing balance(OHAUS), Fourier Transform Infrared Spectroscopy (FTIR) (NICOLET IS 10), Mortar and pestle, n-Hexane, Plastic sieve, Sample bottles, Scanning Electron Microscope (SEM)(JEOL JSM-6480LV), Soxhlet extraction apparatus, Stop watch, Tray, Watermelon seeds, X-ray Fluorescence (XRF)(Xsupreme 800).

2.2 METHODS

2.2.1 Acquisition and Processing of Watermelon (citrulluslanatus) Seed

The seeds were obtained from Muda Lawal market in Bauchi, Bauchi State, Nigeria. They were thoroughly washed and sun-dried for six days after whichthey were shelled and crushed with a mortar and

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pestle, and later dried in an oven at 70°C until constant weight. This was stored in sample bottles. 225g of the crushed and dried seeds were taken and it wascharged for oil extraction using the soxhlet extraction apparatus. About 750ml of n-hexane was used to extract oil from the crushed seeds. The apparatus was left running until the extraction was completed (5hrs50min). The residue was washed with distilled water severally until no trace of n-hexane present. The washed residue was weighed and oven dried in an oven at 70°C until constant weight was achieved. The dried residue (cake) was again pounded and sieved through microns and was stored in sample bottles.

2.2.2 X-Ray Fluorescence.

X-supreme 800 X-Ray Spectrometer was used, which is an energy dispersive microprocessor controlled analytical instrument designed for detection and measurement of elements in a sample (solids, powders and liquids). Sample for X-ray fluorescence analysis were prepared by pressing powdered samples into cellulose before analysis. The prepared sample was loaded in the sample chamber of the spectrometer. Maximum voltage of 30 keV and a maximum current of 1mA were applied to produce the X-rays that excite the sample for a pre-set time (10mins in this case).

2.2.3 Fourier Transform Infrared Spectroscopy

FTIR spectra were taken on a NICOLET IS 10 spectrophotometer using the KBr pressed disk technique. The potassium bromide (KBr) cell was washed with ethanol and cleaned and dried. 150mg grinded samples was weighed, spread on the potassium bromide cell and then placed at the light chamber of the spectrophotometer. The absorption spectrum of the resulted mull was obtained from three minutes scan time in the region between 4000–400cm^{-1}.

2.2.4 Scanning Electron Microscopy Analysis

The coagulant was sprayed on to an aluminium slip. The samples were given a conductive coating (of about 600Å thick) using sputter ion coater and examined with scanning electron microscope (JEOL JSM-6480LV) equipped with a backscattered electron detector for imaging. A focused electron beam was scanned over the sample in parallel lines. The electrons interact with the sample, producing an array of secondary effects, such as back-scattering, that can be detected and converted into an image. The image can then be digitalized and presented to an image analyser, which uses complex algorithms to identify individual particles and record detailed information about their morphology.

3.0 RESULTS AND DISCUSSIONS

3.1 FTIR ANALYSIS

The frequency assignment approach was used for the interpretation of the spectra. Figure 1 shows the FTIR spectrum of the crushed watermelon seed and peaks indicated were assigned to various functional groups.

The frequency bands between 3854.66 and 3802.33 cm^{-1} region corresponds to -OH stretching (water and hydroxyl group). The presence of -OH stretching (water and hydroxyl group) was probably due to the KBr pressed disk technique used in preparing the sample which happens to be a hygroscopic material. The frequency bands between 3752.23 and 3736.41cm^{-1} corresponds to the AlO–H which falls between 3650 and 3770 cm^{-1} was due to AlO–H stretching vibration. The frequency band at 3447.68 cm^{-1} was assigned to N-H group stretching. The bands at 2925.49 and 2854.13 cm^{-1} corresponds to CH_2 asymmetric stretching vibration and CH_2 symmetric stretching vibration respectively, the band at 2930 and 2820 cm^{-1}, which have been found to be sensitive to changes in the gauche/trans conformation ratio and the chain-chain interaction.

The frequency band at 2360.54 cm^{-1} was assigned to Si-H (silane). 1845.28 cm^{-1} is assigned to C=O (5 membered β-lactones) and 1740.89 cm^{-1} was assigned C=O (Carboxyl). The frequency bands at 1654.27 is attributed to –C=C– symmetric stretching of alkenes and 1647.87 cm^{-1} was found to be attributed to N-H bend

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(1^{o} Amines). The bands at 1559.49 and 1541.34 cm^{-1} were assigned to N-H (secondary amides), 1457.99 cm^{-1} was assigned to α-CH_2 bending alkane, and 1419.62 cm^{-1} was assigned to C-N stretch amide III band, and 1242.88 cm^{-1} was assigned to Si–CH_3. The 1161.15 and 1106.88 cm^{-1} frequency bands are attributed to P=O and 668.49 attributed to C–Br from the KBr pressed disk technique used. Table 1 summarises the functional groups assigned to the crushed watermelon seed extract.

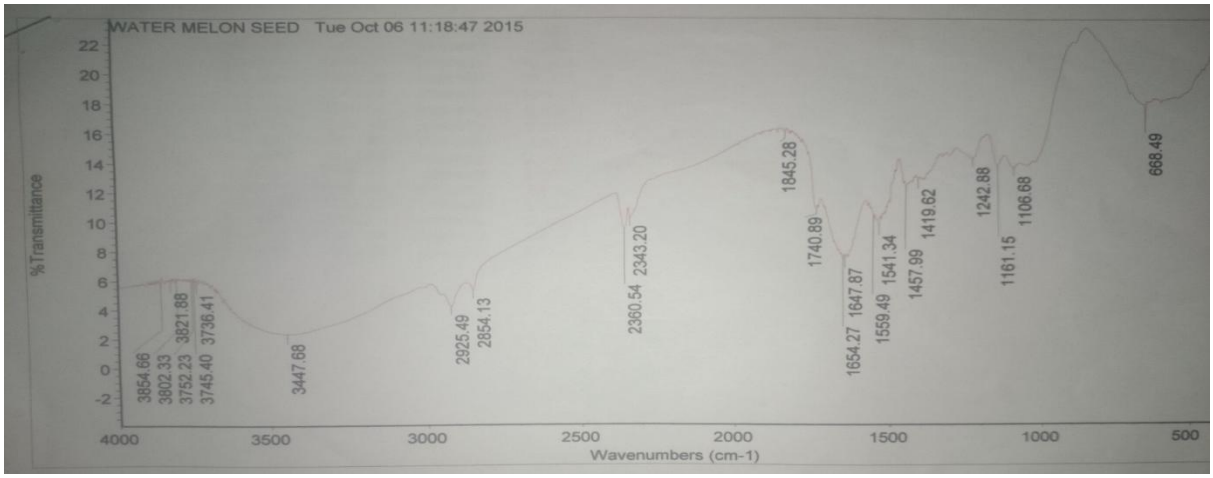

Figure 1: FTIR Spectrum of Crushed Watermelon Seed.

Table 1: Summary of the FTIR analysis result obtained.

Wavenumber (cm^{-1})	Bond	Assignment	Functional groups
3854.66	–OH	Free OH stretch	Hydroxyl & water
3821.88	–OH	Free OH stretch	Hydroxyl & water
3802.33	–OH	Free OH stretch	Hydroxyl & water
3752.23	AlO–H	–OH stretching	Hydroxyl
3745.40	AlO–H	–OH stretching	Hydroxyl
3736.41	AlO–H	–OH stretching	Hydroxyl
3447.68	R–C(O)–NH-RCH_3–I	N-H asymmetric stretching	Amides
2925.49	CH_2–R	Asymmetric stretching	Aliphatic
2854.13	Si-H	Symmetric stretching	Aliphatic
2360.54	C=O		Silane
1845.28	C=O	5 membered β-lactones	Lactones
1740.89	–C=C–	C=O stretching	Carboxyl
1654.27	N-H	Symmetric stretching	Alkene
1647.87	N-H	N-H bend	1^{o} Amines
1559.49	N-H	2¡-amide II band	Amides (secondary)
1541.34	-CH_2	2¡-amide II band	Amides (secondary)
1457.99	C-N	α-CH_2 bending	Alkane
1419.62	Si–CH_3	Amide III band (stretch)	Amides (primary)
1242.88	P=O		Silane
1161.15	P=O	Phosphorus	Phosphate
1106.88	C–Br	Phosphorus	Phosphate
668.49		Alkyl bromide	Alkyl halides

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Natural coagulants are mostly either polysaccharides or proteins. The crushed seed is a protein coagulant, because the seed contains a significant amount of protein (31.9%) (Gwana, et al., 2014). Proteins are large biomolecules, or macromolecules, consisting of one or more long chains of amino acid residues, and protein contains carbon, hydrogen, and oxygen like the carbohydrates and lipids, but also contains phosphorus . Proteins differ from one another primarily in their sequence of amino acids, which is dictated by the nucleotide sequence of their genes, and which usually results in protein folding into a specific three-dimensional (3D) structure that determines its activity . A linear chain of amino acid residues is called polypeptide, and protein contains at least one long polypeptide (Rose, et al., 2006). Short polypeptides, containing less than 20-30 residues, are rarely considered to be proteins, but are commonly called peptides. A peptide bond is an amidecovalent bond formed between the amino andcarboxyl functional groups of separate amino acids(Rose, et al., 2006). The individual amino acid residues are bonded together by peptide bonds and adjacent amino acid residues . Below is the chemical structure of protein polymer. All proteins bind or stick to other molecules . The ability of a protein to bind to other molecules depends on the formation of a set of weak, non-covalent bonds, ionic bonds, and van der Waals attractions and favourable hydrophobic interactions since protein is hydrophobic .

3.2 XRF ANALYSIS

TABLE 2 SHOWS THE XRF RESULT OF THE CRUSHED WATERMELON SEED EXTRACT. THIS GAVE THE AVERAGE ELEMENTS CONCENTRATION IN THEIR OXIDES.

Table 2: Elements Present in the Crushed Seed

ELEMENTS (Oxide)	CONCENTRATION (wt %)	ELEMENTS	WEIGHT (wt %)
Al_2O_3	20.309	Al	10.746
SiO_2	12.44	Si	5.811
P_2O_5	7.06	P	3.078
Fe_2O_3	6.556	Fe	4.581
Na_2O	3.63	Na	2.693
K_2O	2.919	K	2.423
MgO	2.5	Mg	1.508
CaO	0.86	Ca	0.615
TiO_2	0.6	Ti	0.36

The elements present in the crushed watermelon seed in the order of concentration are Al_2O_3, SiO_2, P_2O_5, Fe_2O_3, Na_2O, K_2O, MgO, CaO, and TiO_2. The elements present in the crushed seed that gave it the coagulant properties are Al_2O_3, SiO_2, and Na_2O. Al_2O_3 is the major element in the crushed seed followed by SiO_2. The two major elements Al_2O_3.SiO_2 together gave the crushed seed the ability to cause the precipitation of the particles in the raw water as in the case of bentonite when used as coagulant aid which is composed of Al_2O_3.SiO_2. Also SiO_2 and Na_2O in the crushed seed helps in the addition of weight and size to particles in the raw water as in the case of activated silicate as a floc aid. Standard alum consisted of 17-18 wt% Al_2O_3 and 0.4-0.7 wt% Fe_2O_3.

3.3 Characterization using Scanning Electron Microscopy (SEM)

Below are the backscattered detector (BSD) images of the crushed watermelon seed obtained using Scanning Electron Microscopy (SEM). Figure2 shows the BSD image of 54 particles by count in x245 magnification. As it was prepared as fine ground powder, the morphology shows that the specimen is in particle structures. Figure 3 shows the BSD image of the surface of the particles in x1500 magnification.

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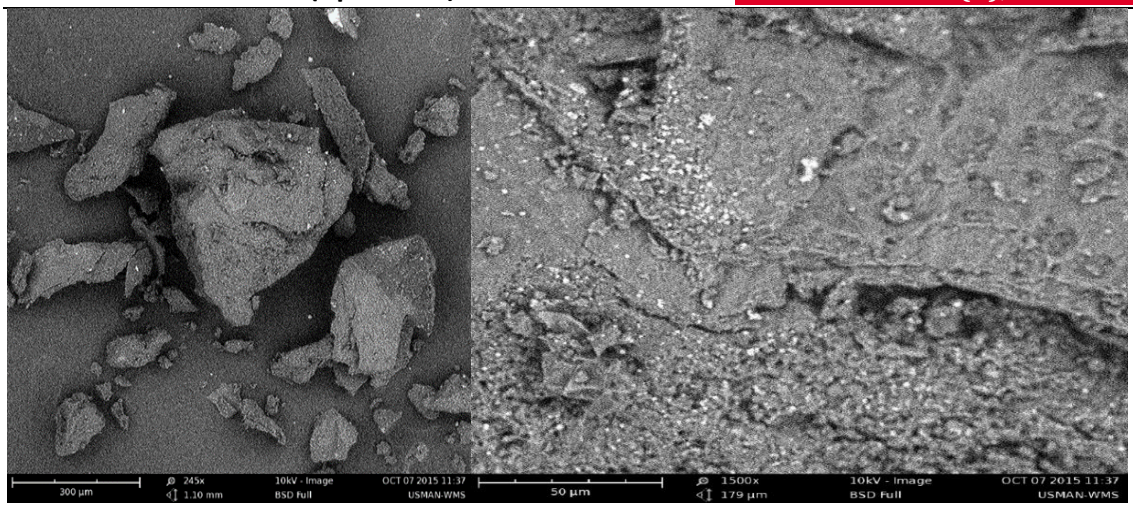

Figure 2: BSD image of 54 particlesFigure3: BSD image of the surface of the particles

In figure 3 above, the surface of the particle is rough. Also the white coloured spots on the image indicates regions with strong intensity and the other grey colours indicates light intensity. Table 3 presents the data of morphological parameters obtained from the SEM image.

Table 3:Data of Morphological Parameters Obtained

Properties	Median	Average
Circle equivalent diameter	53 µm	72.2 µm
Major axis	71.1 µm	95.7 µm
Minor axis	39.1 µm	55.3 µm
Convex hull	208 µm	275 µm
Area	2.21E+03 µm²	5.93E+03 µm²
Circularity	0.547	0.542
Convexity	0.883	0.871
Greyscale	105	106
Aspect ratio	0.576	0.597

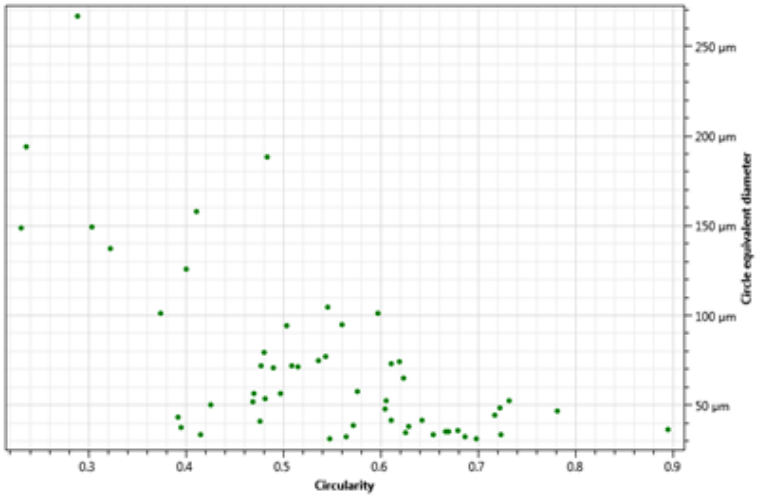

Figure 5: Graph of Circle equivalent diameter and Circularity.

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The graph above represent the relationship between circularity and circle equivalent diameter of the particles of the crushed seed. The graph indicates the shape of the particles and the diameter of the circles with the same area as the particles. Majority of the particles have circularity values in the regions between 0.36 and 0.78 and circle equivalent diameter between 30 and 190 µm. Majority of the particle shapes are: twinning, 4-point stars, 5-point stars, star, torpedo, ribbon, long rectangle, medium rectangle, medium ellipse, touching circles, long ellipse, and equilateral triangle.

4.0 CONCLUSION

Crushed watermelon seed was characterized using FTIR, XRF and SEM. The following conclusions can be deduced from the analyses:

- The FTIR spectra vibrational frequencies of the crushed watermelon seed reveals the functional groups that made up the watermelon seed andalso the following spectra 3447, 1845, 1740, 1647, 1559, 1541, & 1419 revealed the presence of coagulant proteins in the crushed watermelon seed.
- The XRF analysis of the crushed watermelon seed reveals the element present in it that gave it the coagulant properties which are Al_2O_3, Fe_2O_3, SiO_2, and Na_2O.
- The SEM shows the backscattered detector (BSD) images of the crushed watermelon seed shows that the specimen is in particle structures as it was prepared as fine grinded powder, also the surface of the particle is rough, and the white coloured spots on the image indicates regions with strong intensity and the other grey colours indicates light intensity.

REFERENCES

[1]. Beltran-Heredia, J., Sanchez-Martýn, J., & Solera-Hernandez, C. (2009). Anionic surfactants removal by natural coagulant/flocculants products. *Ind Eng Chem Res.*, vol. 48:5085–5092.

[2]. Breiten, B., Lockett, M., Sherman, W., Fiyota, S., Al-sayal, M., Lange, H., . . . Whitesides, G. (2013). Water networks contribute to enthalpy / entropy compensation in protein-ligand binding. *J. Am Chem. Soc.*, 135(41): 15579-15584.

[3]. Chaudhuri, M., & Babu, R. (2005). Home water treatment by direct filtration with natural coagulant. *J Water Health*, vol. 3, pp 27–30.

[4]. Cooke, M. (1871). Strychnos potatorum. *American Journal of pharmacy*, vol. xliii, 1-4, pp12.

[5]. Dobson, C. (2000). The nature and significance of protein folding. In Pain RH ed. Mechanisms of protein folding. Oxford. *Oxford, Oxfordshire: Oxford University press*, 1-28.

[6]. Eric, O. (2011). Particle shape factor and their use in image analysis –part 1. *Theory. Journal of GXP compliance*, Eric Olson (2011). Particle shape factor and their use in image analysis –part 1: Theory. Journal of GXP compliance Vol. 15, (3), 1-6.

[7]. Ghebremichael, K., Gunaratna, K., Henriksson, H., Brumer, H., & Dalhammar, G. (2005). A simple purification and activity assay of the coagulant protein from Moringa oleifera seed. *Water Res.*, vol. 39, pp. 2338–2344.

[8]. Gwana, A., Bako, M., Bagudu, B., Sadiq, A., & Abdullahi, M. (2014). Determination of physicochemical, vitamins, minerals, and proximate composition of varieties of watermelon seed cultivated in Borno State, North-East, Nigeria. *International Journal of Nutrition and Food Sciences*, 238-245.

[9]. Moramudaii, M., & Fernando, P. (2001). Use of seeds of Moringa oleifera to clarify turbid waters and wastewaters. *Vidyodaya J. of Sci.*, vol. 10, pp. 167-182.

[10]. Muhammad, I., AbdulSalam, S., AbdulKarim, A., & Bello, A. (2015). Watermelon seed as a potential coagulant for water treatment. Global Journal of Researchers in Engineering: Chemical Engineering. *Chemical Engineering. Global Journals Inc. (USA)*, Vol. 15, 1, pp 17-25.

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[11]. Nelson, D., & Cox, M. (2005). *Lehinger's principles of biochemistry.* New York: WH Freeman and company.

[12]. Rose, G., Fleming, P., Banavar, J., & Ritan, A. (2006). A backbone-based theory of protein folding. *Proc. Nat. Acad. Sci. USA*, 103(45). 18823-18833.

[13]. Santos, A., Luz, L., Argolo, A., Teixeira, J., Paiva, P., & Coelho, L. (2009). Isolation of a seed coagulant Moringa oleifera lectin. *Agricultural Research*, vol. 1(3), pp. 065-069.

[14]. Talbot, B., Narasiah, K., & Ndabigengesere, A. (1995). Active agents and mechanism of coagulation of turbid waters using Moringa oleifera. *Water Res.*, vol.29, pp. 703–710.

[15]. Thomas, .., & Jurgen, B. (2002). Phosphorus reduction in a shallow hypereutrophic reservoir by in-lake dosage of ferrous iron. *Water Res.*, vol. 36, pp. 4525–4534.

[16]. Yang, X., Lu, Z., & Chen. (1999). Spectroscopic study of the aluminium specification in removing humic substances by Al coagulation. *Water Res*, vol. 33, pp. 3271–3280.

[17]. Zhang, F., Zhang, Y., & Luo-Yang, H. (2006). A preliminary study on cactus as coagulant in water treatment. *Process Biochem.*, vol. 41, pp. 730–733.