



# Effect of Alum Treatment on Functional Properties of Cassava Starch

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## **Authors' contributions**

*This work was carried out in collaboration between both authors. Author MOA designed the study, managed the analyses of the study and wrote the first draft of the manuscript. Author OAA managed the analyses of the study and managed the literature searches. Both authors read and approved the final manuscript.*

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## **ABSTRACT**

Chemical modification is usually carried out to overcome the unstable properties of plant starches and improve their physical and functional characteristics during processing. Alum modification at different concentrations (1.0%, 2.0%, 3.0%, 4.0% and 5.0% w/v) with and without 10ml of 1M sodium hydroxide at room temperature was carried out. Cassava starch treated with 3.5% of alum showed a profound influence on pasting properties compared to the native starch in that it exhibited a reduction in the peak viscosity of the modified cassava starch increased. Alum modification also freeze-thaw stability.

**Keywords:** Cassava; alum; functional properties; modification; native starch.

## **1. INTRODUCTION**

In nature, starch is available in an abundant quantity as a naturally occurring organic

carbohydrate. Native starches have been widely used in many industries. Nevertheless, due to the improvement of production technology and continuous development of new products, stricter

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demand for starch properties and suitability are required. Consequently, it is necessary to modify starch properties to provide improved application, suitability and functionality. The modification methods in practice are physical, chemical and enzyme conversion through links cleavage, reform or oxidation [1,2]. Cassava starch has many remarkable characteristics including high paste viscosity, high paste clarity and high freeze-thaw stability which are advantageous to many industries. Modification of cassava and other plant starches using various chemical reagents such as Lactic acid, ammonium phosphate, sodium acetate, sodium acetate with adipic acid, sodium acetate with fumaric acid have been reported by several workers [3,4,5,6, 7,8].

When these organics acids are mixed with flour, it will produce distinctive aroma and taste which is different from the native cassava flour flavour that is preferred by consumers [9].

Substitution of these starches is often accomplished at high temperatures above 100°C using large amounts of reagents and for very long periods. Besides, the volatile nature of some of the reagents e.g acetic anhydride usually necessitates elaborate safety precautions with a consequent increase in production costs. As a result, it becomes imperative to source for an alternative means which would be available in abundant quantity and inexpensive for modification. Alum, a common and cheap chemical compound has been reported to be a potential modifying agent for rice starch [10]. This paper reports results of the effect of alum treatment on functional properties of cassava starch.

## 2. MATERIALS AND METHODS

### 2.1 Materials

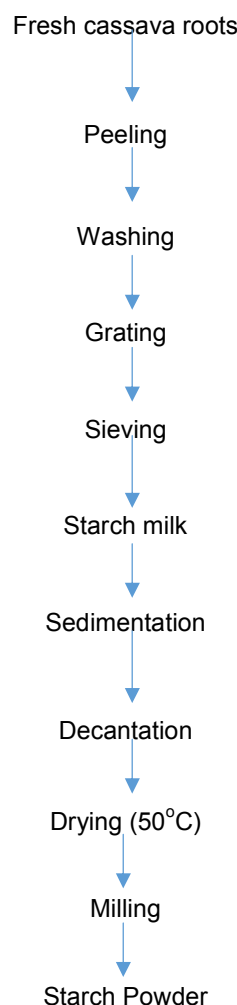
Low cyanide and high yield cassava roots of clone TMS 30572, aged 12-months at harvest were obtained from International Institute of Tropical Agriculture (IITA), Ibadan. Aluminium Sulphate  $[Al_2(SO_4)_3 \cdot 10H_2O]$  i.e Alum was obtained from the laboratory of the water treatment plant, University of Ibadan. Sodium hydroxide used was of analytical grade.

### 2.2 Preparation of Alum Treated Cassava Starch

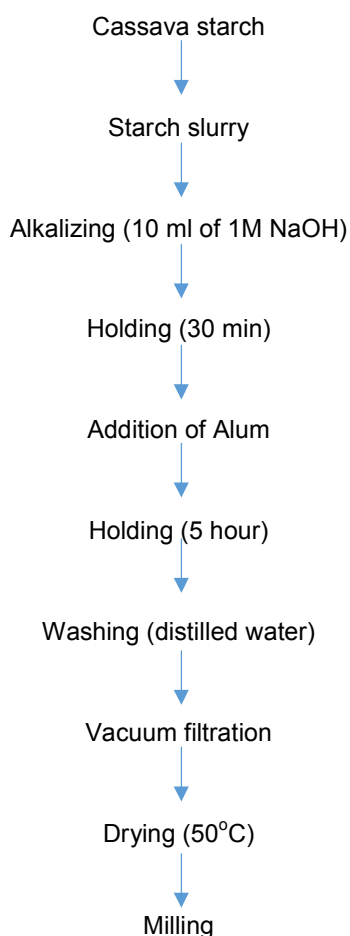
The method described by Shin-Young et al. [10] was used for the preparation of modified cassava

starch as illustrated in Fig. 1. Three samples (150g each) of the native starch were suspended in 200ml of distilled to form a slurry at room temperature to which 10ml of 1M NaOH was added to adjust the pH. The aqueous alkaline suspended was then held for 30min at room temperature with occasional stirring.

Alum was added to the two batches at concentration of 1.0%, 2.0%, 3.0%, 4.0% and 5.0%W/V and were held for 5 hours. At the end of the holding period, samples were washed with about 600ml of distilled water through a Whatman No. 4 filter paper under vacuum suction. The resultant modified starches were dried at 50°C in the oven, cooled at room temperature and subsequently milled into powders and packaged in airtight containers.



**Fig. 1. Flowchart for the extraction of cassava starch**



**Fig. 2. Flowchart for the preparation of Alum modified starch**

## 2.3 Proximate Analysis

The moisture content and Ash content of the modified cassava starch was determined using [11] method. The pH of the starch was determined using a pH meter model 702.

## 2.4 The Degree of Sulphate Substitution

Uv-visible spectrophotometer (Systronics, India) at 490 nm was used to measure the degree of sulphate substitution.

## 2.5 Paste Properties

Swelling power and solubility index was measured as described by Leach et al. [12]. The procedure of Craig et al. [13] was followed for analysis of paste clarity. Freeze-thaw stability was determined by the method of [14,7].

## 2.6 Pasting Characteristics

The pasting characteristics of the starch sample were examined with a Rapid Visco Analyser (model RVA-super 4, Newport Scientific Perten Instrument AB, Huddinge, Sweden). The starch slurry (2.5g with 25 ml distilled water) at pH 6.5 pasted at a heating rate from 50-95°C, cooled from 95°C to 50°C and finally held at 50°C for 30 min. The following measurement was taken from the RVA curve; the pasting temperature, the peak viscosity during the heating stage, the consistency at 95°C, the consistency at 50°C, the setback viscosity calculated as the viscosity of paste when cooled to 50°C minus the peak viscosity.

## 3. RESULTS AND DISCUSSION

The percentage recovery of alum modified starch range from 95.6%-98.0%. This value is much higher because of the slurry method used compared with the percentage recovery obtained by reflux method [5].

The swelling power of alum modified cassava starch with sodium hydroxide increased (11.5% and 16.1%) as the quantity of alum increases, while the swelling power of the modified starch without sodium hydroxide decreased (7.8% and 9.8%). The swelling power of the native starch was 10.0%. The above difference in swelling power shows a modification of the cassava starch. The swelling of the modified starch that contains sodium hydroxide reflects the interaction of hydrolysed amylose chain with water molecules. This observation is in agreement with hydroxypropylated of rice starch [8]. The starch was soluble when heated at temperature of 80°C. The level of solubility corresponds to the concentration of alum used for modification, the higher the level of alum used the higher the percentage solubility (15.1% to 20.0%). A similar result has been reported using HCl [15,10,16].

The apparent viscosity of the pretreated modified starch decreases as the level of alum added increases. The lowest apparent viscosity was found at 5.0% alum concentration. A similar result was reported by '10' on gelatinised waxy rice starch. The apparent viscosities (10-20cp) of the modified cassava starches were lower compared to that of the native starch (22cp). The difference in apparent viscosity of both the modified cassava starches was not significant. The increase in apparent viscosity may be as a

result of the substitution of a sulphate group on the hydrolysed starch molecule. This substitution prevents re-association of the unit molecule in starch. The lower apparent viscosity was found at 5.0% alum concentration.

The reaction efficiencies of alum modified cassava starch with and without sodium hydroxide at different alum concentration were indicated by the degree of substitution of the sulphate group into granular starches. Native cassava starch was presented as zero degrees of substitution since it was used as a blank for treated starches. Different degree of substitution values was found since the cassava starches were modified with different level of alum (1.0% to 5.0%) which ranged from 0.010 to 0.143. out of three possible substitutions for every D-glucopyranosyl unit, respectively. The concentration at 4.0% and 5.0% showed a significantly higher degree of substitution, which enabled sulphate groups to produce the distarch sulphate derivative. Therefore cassava starch modified with a high level of alum showed a significantly higher sulphate content than the native starch. The degree of substitution at a low level of alum (1.0% and 2.0%) results in a low level of degree of substitution. This is desirable because substitution of starches for use in foods need not be at a very high level.

The pasting properties of the alum modified and native starch is presented in Table 2. The result obtained illustrates that the viscosity of the modified starches reduced but there was no chronological order as compared to that of native starch. Therefore, the decrease in viscosity breakdown can be indicative that modification has taken place. This is most likely due to the effect of substitution in the hydroxyl group by sulphate group from the alum used and that the glucosidic bonds in the starch are hydrolysed. Osunsanmi et al. [5] and Olatunji [17] reported reduced viscosity curves at lower pH values for waxy corn starch.

The pasting temperature tended to increase. This was another indication that substitution had taken place inside the starch granules. Therefore, the treatments increased resistance to heat and shear as applied in the Rapid Visco Analyser. This result is in agreement with the report made by Olatunji [17] on acetate and citrate substituted cassava starch.

The setback viscosity increased for the alum modified starch as compared to that of the native

starch. The granular integrity under the heat and shear conditions applied in the Analyser and would consequently show a higher viscosity development during the cooling period, resulting in a higher setback. Retrogradation has been attributed to the linear fraction of molecular starch (amylose) and the introduction of substituent group interfere with re-alignment of the linear fractions and consequently reduced manifestation of retrogradation.

It has been known that native cassava starch increased as freeze-thaw cycles were increased. Figs. 3,4 & 5 illustrates the significant effects of alum modification at different levels for improving freeze-thaw stability. The order of freeze-thaw stability of the alum modified starch with and without sodium hydroxide has a close relationship with increasing degree of substitution. Alum modification introduces monofunctional sulphate groups to the hydroxyl group of the starch molecules, thus preventing dissolved linear starch molecules from associating closely by reduction of the attractive forces between hydroxyl groups on adjacent chains during cooling or freezing. Therefore, the higher the alum contents in terms of the degree of substitution, the lower the syneresis. This result of improved freeze-thaw stability for alum modified starch as the concentration of alum added increases was similar to the result obtained [17] for substituted starches of acetate and hydroxypropylation of sago starch.

**Table 1. Proximate composition of Native Starch**

	% Composition
Starch	87.6
Protein	0.5
Ash	0.2
Fibre	-

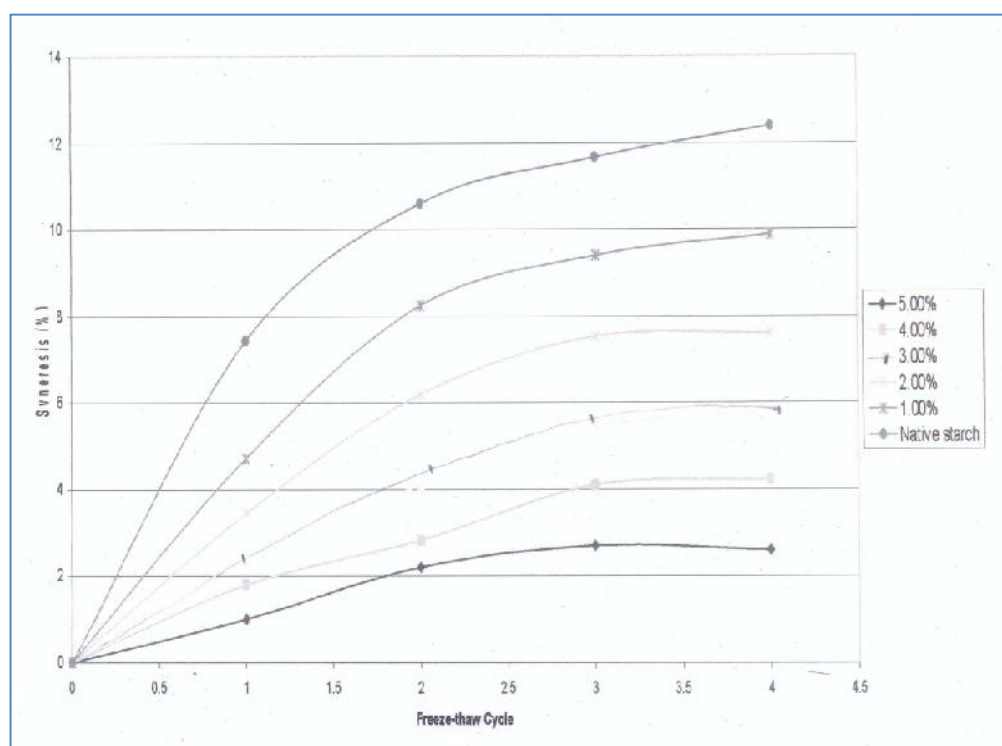
The percentage transmittance of paste clarity of the starches was presented in Table 2. The percentage transmittance of the alum modified starch pretreated with sodium hydroxide (34.1% to 43.4%). The paste clarity of the alum modified cassava starch showed a decreased percentage transmittance (29.4% to 32.7%). In this study, therefore, paste clarity was used to compare the efficiencies of sodium hydroxide. The alum modification of starch can be confirmed by the reduction of paste clarity [6,7]. The result obtained corresponds with the result obtained for swelling power.

**Table 2. Properties of Alum modified and Native Cassava starch**

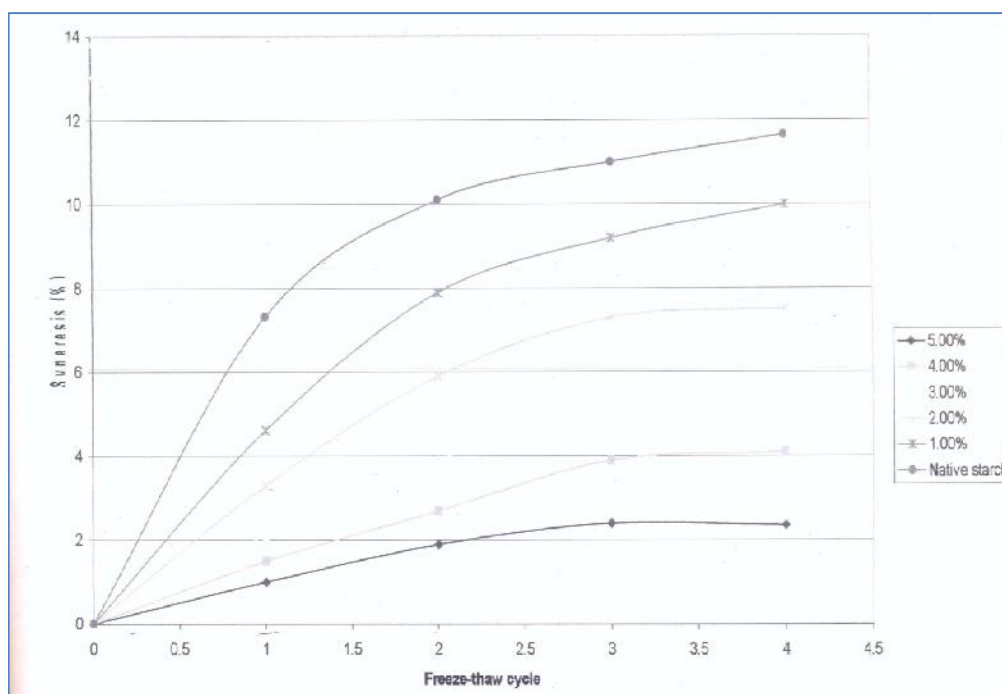
Conc. of Alum in starch sample	% recovery	Swelling power %	Solubility %	Apparent Viscosity (cp)	Degree of Substitution	Paste clarity (% T <sub>620</sub> )
1.0	96.0	9.8	15.1	18.0	0.010 ± 0.001	32.1
2.0	96.9	9.2	16.0	17.0	0.016 ± 0.001	31.3
3.0	97.7	8.8	18.7	15.0	0.023 ± 0.001	30.6
4.0	97.9	8.0	19.6	13.0	0.034 ± 0.001	29.6
5.0	98.0	7.8	19.9	11.0	0.043 ± 0.001	29.4
<b>10ml 0.1M NaOH</b>						
1.0	95.6	11.5	15.2	20.0	0.010 ± 0.001	34.1
2.0	96.0	12.7	17.8	19.0	0.016 ± 0.001	36.0
3.0	97.3	13.8	18.5	14.0	0.023 ± 0.001	38.3
4.0	97.5	15.8	19.8	12.0	0.034 ± 0.001	40.7
5.0	97.9	16.1	20.0	10.0	0.043 ± 0.001	43.4
Native starch	N.A	10.0	12.8	22.0	0.000 ± 0.000	33.33

*N.A – Not Applicable***Table 3. Pasting properties**

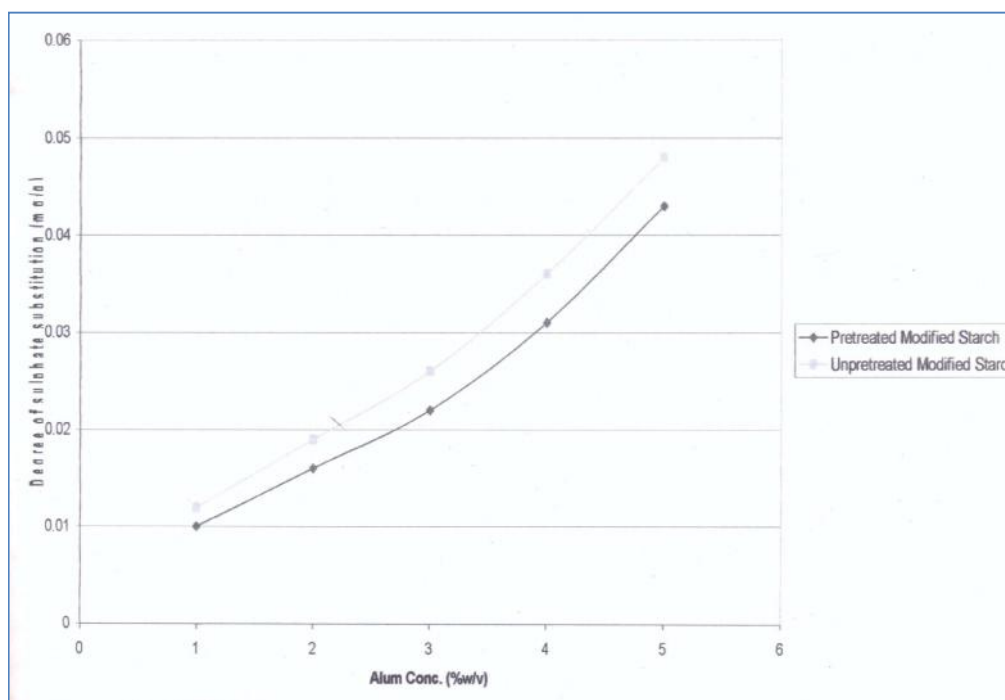
Conc. of Alum in starch (%)	Peak Viscosity (RVU)	Trough (RVU)	Breakdown Viscosity (RVU)	Final Viscosity (RVU)	Setback Viscosity (RVU)	Peak Time (Mins)	Pasting Temp (°C)
5.0	307.42	120.17	187.25	179.08	58.92	4.00	74.35
4.0	301.50	118.33	183.17	163.17	44.83	4.00	74.25
3.0	347.58	137.50	234.83	208.75	73.33	4.00	75.05
2.0	325.00	133.92	191.08	173.25	39.33	4.00	73.55
1.0	347.42	132.00	215.42	185.83	53.83	4.00	73.55
<b>With 10ml 0.1M NaOH</b>							
5.0	279.08	75.08	204.00	127.83	52.75	3.87	75.25
4.0	294.42	82.83	211.58	137.83	55.00	3.87	75.15
3.0	293.83	122.33	171.50	176.92	54.58	4.07	75.25
2.0	281.08	82.83	188.58	136.33	53.50	3.87	75.05
1.0	345.67	157.08	215.42	190.83	33.75	4.00	73.55
<b>Native Starch</b>	356.67	112.67	244.00	137.67	25.00	3.87	73.45



**Fig. 3. Freeze-thaw Stability of pretreated alum modified and native Starch**



**Fig. 4. Freeze-thaw Stability of treated alum modified and native Starch with 10ml 0.1N NaOH**



**Fig. 5. Effect of Alum concentration on degree of sulphate substitution**

#### 4. CONCLUSION

Results obtained showed that alum modification improves the physical and functional properties of cassava starch at room temperature under the condition of pretreatment with and without sodium hydroxide. Varying the concentration of alum affected the extent of modification. Better percentage recovery was obtained for the modified starches. It was found that the swelling power of the alum modified cassava starch pretreated with sodium hydroxide increased which was more efficient while modified starch without sodium hydroxide decreased. The solubility or hydrophobicity of modified starches was improved which will enhance better digestibility. The degree of substitution was used to show the efficiency of alum for modification. Other improved properties are apparent viscosity, paste clarity, pasting characteristic and cooking stability. The modified starch can be incorporated into foods where such functional properties are desired such as thickening agents, frozen foods, canned foods e.t.c. Under those conditions of alum modification, undesirable starch properties were counteracted.

It is recommended that the freeze-thaw cycles should be increased and samples have gone through Rapid Visco Analyser should be used.

The alum modified cassava starch should be used in the processing of food to determine the best suitability in the processing industry.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

#### REFERENCES

1. Jane J. Structural aspects of starch granule In: Starch '96- the book, perspectives for a versatile raw material on the threshold of a new millennium; 1997.
2. Joko S, Kazuhiko N. Physicochemical properties of modified cassava starch prepared by application of mixed microbial starter. International Journal of Research In Agriculture and Food Sciences, 2012;2(7).
3. Akpa J. Gunorubon, Dagde KK. Modification of cassava starch for industrial uses. International Journal of Engineering and Technology. 2012;2(6).
4. Elisa J, Zulkifli L, Ridwansyah Era, Y, Ismed S. Physicochemical and functional properties of fermented starch from four cassava varieties. Asian Journal of

5. Osunsanmi AT, Akingbala JO, Oguntimehin GB. Effect of storage on starch content and modification of cassava starch. *Starch/Starke*. 1989;41(2):54-57.
6. Lim S, Seib PA. Preparation and pasting properties of wheat and waxy corn starch phosphates cereal chemistry. 1993;70: 137-144.
7. Wu Y, Seib PA. Acetylated and hydroxyl propylated di starch phosphates from waxy barley paste properties and freeze thaw stability cereal chemistry. 1990;67:202-208.
8. Yeh An, Yeh Su-Lan. Some characteristics of hydroxyl propylated and cross-linked rice starch. *Cereal Chemistry*. 1993;70(5): 596-601.
9. Subagio A. Standard operating procedures on cluster based production of Mocaf. National Competitive Research, Staple Food Diversification. SEAFST Center. IPB, Bogor; 2008.
10. Shin-Young Lee, Sang-Gui L, Ik-boo Kwon. Effect of alum on the rheological properties of gelatinized solutions of non-waxy and waxy rice starches. *Korea Journal of Food Science and Technology*. 1995;27(5):776-782.
11. AOAC. Official methods of analysis 22nd edition. Association of Official Analytical Chemist. Washington. D.C; 2005.
12. Leach HW, Mc Cowen LD, Schich TJ. Structure of the starch granule. I. Swelling and solubility patterns of various starches. *Cereal Chem*. 1959;36:534-544.
13. Craig SAS, Mainngat CC, Seib PA, Hosonet RC. Starch paste clarity. *Cereal Chemistry*. 1989;66:173-174.
14. Takashashi S, Maningat CC, Seib PA. Acetylated and hydroxypropylated wheat starch: Paste and gel properties compared with modified maize and tapioca starches. *Cereal Chemistry*. 1989;66:499-506.
15. Shi YC, Seib PA. The structure of four ways starches related to gelatinization and retrogradation. *Carbohydrate Res*. 1992;227:131-145.
16. Vasanthan T, Bhatti RS. Physicochemical properties of small and large granule starches of waxy, regular and high-amylose barleys. *Cemal Chem*. 1996;73(2):198-207.
17. Olatunji MO. Effect of alum treatment on functional properties of cassava Starch. M.Sc Thesis of the Department of Food Technology, Faculty of Technology, University of Ibadan. Ibadan; 2005.

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