

Full Length Research Paper

# Influence of acetylation on the physicochemical properties of composited starches from sweet potato (*Ipomoea batatas* L.) and water yam (*Dioscorea alata* L.)

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The study investigates the effect of acetylation on the physicochemical properties of composited starches from sweet potato and water yam. Starch was respectively isolated from both sources, dried and subjected to acetylation at different combination. The result shows that the modified starches were of low percentage of acetylation and low degree of substitution. Acetylation increased the water absorption capacity of the starch while compositing itself seemed to have a counteracting influence on the parameter. The oil absorption capacity generally got reduced with acetylation while synergistic role of acetylation and compositing was observed in the swelling capacity of a composited starch (SP/WY-20/80) and counteracting influence in others. The solubility indices of starches were greatly enhanced by acetylation and compositing at all temperatures of evaluation (50-90°C). Acetylation improved the colour lightness (L\*-value) of the starches (93.05-94.02) while that of the native starches ranged between 92.18 and 92.31. Most pasting variables (peak, breakdown, final and setback viscosities) were lower in acetylated composite starches than that of the unmodified counterparts. Freeze-thaw stability of gels from composited starches was greatly enhanced as lower volume of exudate was generated from the acetylated starches in all the freeze-thaw cycles. The findings in this study have the potential of creating awareness among the food industry with respect to acetylated starch production from both sweet potato and water yam.

**Key words:** Acetylation, starch, sweet potato, water yam, physicochemical properties.

## INTRODUCTION

Starch is a commodity that serves as a food ingredient but with a uniqueness of having a greater versatility of application in the food industry than any other single food

ingredient. Starch is obtainable from diverse food material sources such as cereals (Ptaszek and Grzesik, 2007), root and tuber crops (Peroni et al., 2006) and from

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other plants including African fan palm (*Borassus aethiopum*) (Bolade and Bello, 2006) and Christ thorn seed (*Ziziphus spina-christi*) (Izuagie et al., 2012). Starch in its native form has limited usage in the food industry as it is prone to low shear resistance, minimal thermal resistance and greater tendency towards thermal decomposition and retrogradation (Singh et al., 2007). However, native starch is considered as a good texture stabilizer and regulator in food systems (Cousidine, 1982). Starches from diverse biological origins do exhibit variations in their physicochemical properties and functional characteristics and therefore their modifications are usually tailored towards meeting the requirements of specific food applications (Hermansson and Svegmarm, 1996). The starch modification techniques are usually physical (e.g. pregelatinization), enzymatic (e.g. enzymatic hydrolysis) and chemical (e.g. oxidation, etherification, esterification and cross-linking) (Singh et al., 2007).

In Nigeria, the utilization of sweet potato (*Ipomoea batatas* L.) tuber is limited to human food and there is no visible industrial application. The fresh tubers are usually subjected to processing through boiling, roasting, frying or baking while it is occasionally used as an ingredient for making meat pies particularly in the urban areas (Tewe et al., 2003). The use of water yam (*Dioscorea alata* L.) is also limited to human food as against white yam (*D. rotundata* Poir) which has found an increasing use at industrial level (Coursey and Ferber, 1979). Therefore, the production of starches from sweet potato and water yam respectively is a way of expanding their potential application while the modification (acetylation) of their composited starches will serve as a greater way of exploring the possibility of industrial uses of these less-exploited tubers.

## MATERIALS AND METHODS

Freshly harvested sweet potato (*Ipomoea batatas* L.) and water yam (*D. alata* L.) tubers were purchased from a local farmer at Akure, Ondo State, Nigeria.

### Starch extraction

Starch was extracted from sweet potato and water yam tubers respectively following the method of Lawal (2004) with some modifications. Two kilograms each of sweet potato and water yam tubers were respectively peeled, washed and grated at high speed for 2 min in a Warring blender with 1 L of distilled water. The slurry obtained was dispersed in 9 L of distilled water to make a total of 10 L of distilled water used. The mixture was stirred for 15 min before being filtered through a 200-micron screen, passed again through a 100-micron screen and then centrifuged (Eltek centrifuge, MP 400R, Electrocraft, India) at 1500 rpm for 20 min. After removing the mucilaginous layer, the sediment was washed several times by suspension in distilled water and centrifuged again until it appeared to be free of non-starchy material. The sediment was then oven-dried at 55±2°C for 48 h. The oven-dried starch cake was ground, passed through a 75-micron screen and stored in low density polyethylene (LDPE) bags at ambient temperature till further use.

### Formulation of composite starch

Extracted starches from both sweet potato and water yam were composited, thoroughly mixed and sieved together to ensure homogeneous mixing at different combination ratios of 100/0, 0/100, 80/20, 60/40, 40/60 and 20/80 (sweet potato/water yam). Native starches (unmodified) from sweet potato and water yam served as the control. Thereafter, the formulated starches were individually packaged in sealed polyethylene bags and kept at ambient temperature (30±2°C) for subsequent use.

### Acetylation of composited starch

The method of Sathe and Salunkhe (1981) was used for this analysis. The starch sample (100 g) was dispersed in 500 ml of distilled water and stirred magnetically for 20 min. The pH of the slurry obtained was adjusted to 8.0 using 1.0 M NaOH. Acetic anhydride (10.2 g) was added slowly to the mixture while maintaining a pH range of 8.0 to 8.5. The reaction was allowed to proceed for 5 min after the addition of acetic anhydride. The pH of the slurry was finally adjusted to 4.5 using 0.5 M HCl. It was then filtered, washed four times with distilled water and dried in the air oven at 45±2°C for 48 h.

### Determination of acetyl percentage and degree of substitution

The determination of percentage of acetylation (% acetyl) of the starch samples and degree of substitution (DS) was carried out using the modified method of Golachowski (2003). The acetylated starch (10 g, dry basis) was mixed with 65 ml distilled water in a conical flask and neutralized by adding few drops of 0.5 M NaOH to obtain a faint pink colour with phenolphthalein indicator. Twenty-five millilitres of 0.5 M NaOH was added to the mixture and mixed thoroughly for 35 min using magnetic stirrer at 1000 rpm. The resultant mixture was titrated against 0.5 M HCl until the pink colour disappeared and the result calculated. The original unmodified starch was also used for the evaluation.

$$\text{Degree of acetylation (\%)} = \frac{(\text{Blank} - \text{sample}) \text{ml} \times \text{Molarity of HCl} \times 0.43 \times 100}{\text{Weight of sample}} \quad (1)$$

Degree of substitution (DS) is defined as the average number of sites per glucose unit that possess a substituent group.

$$\text{DS} = \frac{[162 \times \text{Acetyl}(\%)]}{[4300 - (42 \times \text{Acetyl}(\%))] } \quad (2)$$

### Determination of swelling power and solubility of starch samples

The swelling power and solubility of the starches were determined by using the method of Leach et al. (1959). Starch (1 g db) was weighed into centrifuge tubes and 50 ml distilled water added. These tubes were immersed in water bath at temperature ranging from 50 to 90°C at 10°C interval for 30 min and thoroughly and constantly stirred with glass rod during the heating period. The tubes were removed, cooled to room temperature and centrifuged (Eltek centrifuge, MP 400R, Electrocraft, India) at 3,000 rpm for 15 min. The supernatant was carefully transferred into a conical flask and 5 ml out of it were pipetted into weighing Petri dishes, evaporated over a steam bath and dried in the air oven at 110°C for 4 h. The weight of the paste was determined and used to calculate the swelling power as gram of sediment paste per gram starch.

**Table 1.** Percentage of acetylation and degree of substitution in the composited starches.

Starch sample <sup>1</sup>	Percentage of acetylation (%)	Degree of substitution (DS)
UNSPS	0	0
UNWYS	0	0
ASPS	1.08	0.041
AWYS	1.11	0.042
SP/WY-80/20	1.13	0.043
SP/WY-60/40	1.15	0.044
SP/WY-40/60	1.11	0.042
SP/WY-20/80	1.13	0.043

<sup>1</sup>Starch sample: UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio.

#### Determination of water and oil absorption capacities

The water absorption capacity (WAC) and oil absorption capacity (OAC) of each starch sample was determined using the method of Sathe et al. (1982). A suspension of 1 g of starch (db) in 10 ml of distilled water or in 10 ml of 'Executive Chef' vegetable oil with density of 0.92 g/ml was initially prepared. The suspension was stirred for 5 min using magnetic stirrer (Model 7664, Stuart Scientific, UK) at 500 rpm. The mixture was then transferred into a centrifuge (MSE minor 35) for 30 min at 820 g. The free water obtained was removed carefully and the volume of the water/oil was determined. The water or oil absorbed by the starches was calculated as the differences between the initial water/oil used and total (g/g) of water/oil absorbed by the starch.

#### Determination of colour characteristics of starch samples

The colour characteristics of the starch samples were measured using a colour measuring instrument (Model SN 3000421, ColorTec-PCM, USA) and the values expressed on the L\*, a\*, b\* tristimulus scale. The standardization ( $L^* = 94.61$ ,  $a^* = 0.62$ ,  $b^* = 14.96$ ) of the instrument was first carried out using a white reference standard (white duplicating paper sheet, 80 g/m<sup>2</sup>). Three grammes (3 g) of the starch sample were put in a clean paper and the colour meter was placed on the sample by allowing the sensor to touch the sample. The reading was taken directly and the results from three replicates per sample were averaged. The colour purity, expressed as chroma (C), was calculated from  $(a^{*2}+b^{*2})^{1/2}$  (McGuire, 1992).

#### Evaluation of pasting properties of starch samples

The pasting properties of the starch samples were evaluated using a Rapid Visco Analyzer, RVA (Newport Scientific Pty. Ltd., Australia). The moisture content of all starch samples was determined and each sample was weighed into the canister at different time to form slurry using distilled water (7%, w/w). Series of operational procedures were followed and a programmed heating and cooling cycle was then used, where the slurry was held at 50°C for 1 min, heated to 95°C at 6°C/min, held at 95°C for 5 min, finally cooled from 95 to 50°C at 6°C/min, and held at 50°C for 2 min. The pasting properties of each sample were inferred from the acquired computer-generated data which include the pasting

temperature, peak viscosity, time to peak, breakdown, holding strength or trough, setback and final viscosity.

#### Evaluation of freeze-thaw stability of gels from the starch samples

The freeze-thaw stability of gels obtained from the starch samples was investigated using the method of Kaur et al. (2004). Aqueous suspension 5% w/v (db) of each starch sample was prepared using distilled water. The suspension was heated to 95°C for 30 min in water bath and then cooled with continuous stirring to prevent skin formation. The paste was subjected to alternate freezing and thawing (18 and 3 h, respectively for five cycles). This was centrifuged at 920 rpm for 10 min and percentage exudates was determined and plotted against the number of freeze-thaw cycle.

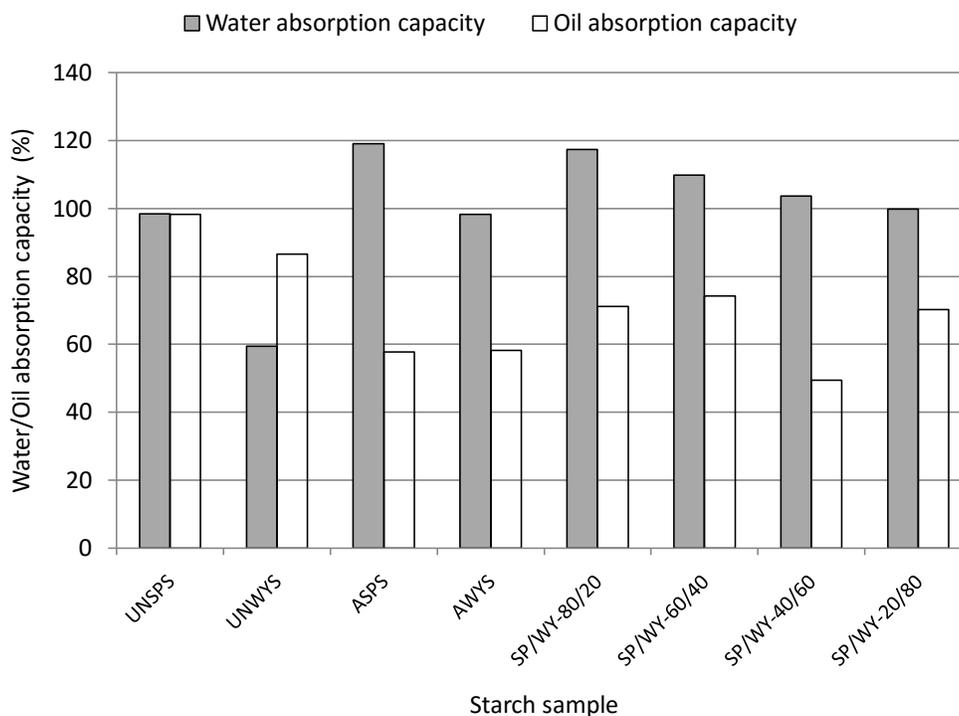
#### Statistical analysis

All determinations carried out in this study were done in triplicates. A mean value and standard deviation were calculated in each case. Analysis of variance (ANOVA) was also performed and separation of the mean values was by Duncan's multiple range test at  $p < 0.05$  using Statistical Package for Social Scientists (SPSS) software, version 16.0.

## RESULTS AND DISCUSSION

#### Percentage of acetylation and degree of substitution in the composited starches

The percentage of acetylation in the composited starches (Table 1) showed a range of 1.08 to 1.15%; with samples ASPS and SP/WY-60/40 giving the lowest and highest values, respectively. Marginal differences were observed in the percentage of acetylation which implies that the level of acetylation reaction in the starch samples was almost the same. Percentage of acetylation in a starch sample has been shown to be a function of reaction time and ratio of acetic anhydride to starch (Xu et al., 2004).



**Figure 1.** Water/oil absorption capacity of composited starches as influenced by acetylation. UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio.

For food-grade acetylated starches, the maximum limit set for percentage of acetylation by the United States Food and Drug Administration (USFDA) is 2.5% acetyl content (Thomas and Atwell, 1997).

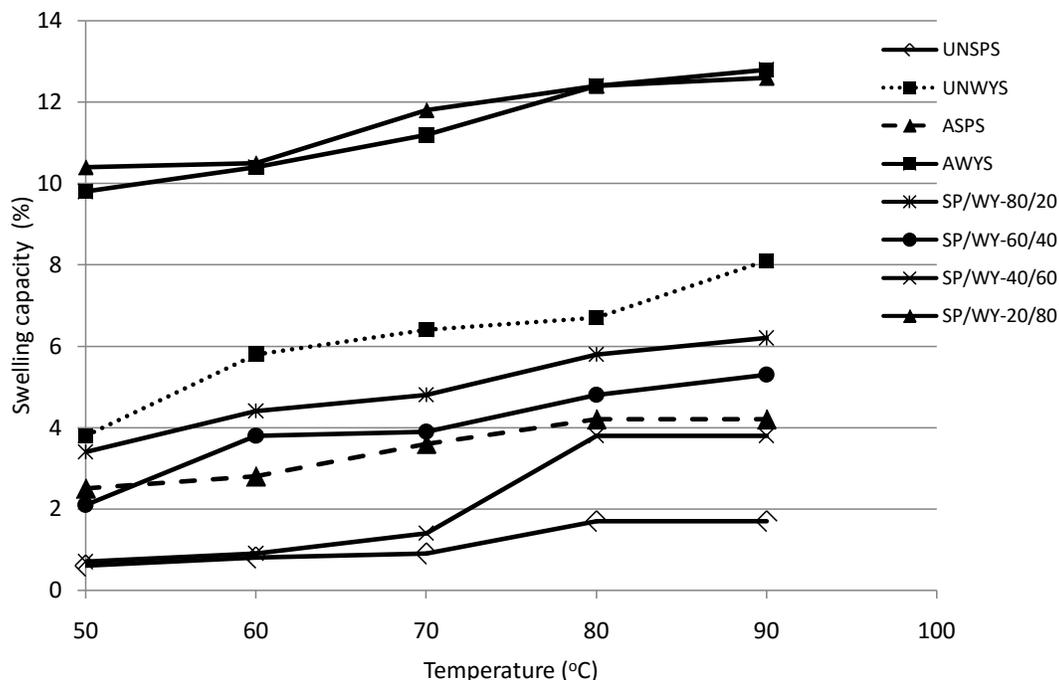
The degree of substitution (DS) in the composited starches (Table 1) also showed a range of 0.041 to 0.044, which implies low values and marginal differences. The DS essentially represents the average number of sites per glucose unit that possess a substituted acetyl group within the starch molecules (Mirmoghtadaie et al., 2009). The low value of DS in an acetylated starch generally has been attributed to such factors as lack of granular surface pore or enough large inner channels which facilitate physical access of acetic anhydride to the interior of the starch granules (Gonzalez and Perez, 2002).

#### **Water and oil absorption capacities of composited starches as influenced by acetylation**

The water and oil absorption capacities of the composited starches as influenced by acetylation are presented in Figure 1. The water absorption capacity (WAC) of the starch samples ranged between 59.4 and 119.1% with

UNWYS and ASPS having the lowest and highest values, respectively. The acetylation process generally led to the increase in the water absorption capacity of the modified starches. The general increase in WAC can be attributed to lower values of percentage of acetylation observed in this study which might have facilitated an increase in water percolation and retention within the starch granules than the native starches due to structural re-organisation of the starch molecules (Jarowenko, 1986). However, certain previous findings (Biliaderis, 1982) had observed that acetylation could lead to reduction in WAC of waxy maize starch. This can be attributed to the introduction of hydrophobic group into the starch molecules which in turn is dependent on the percentage of acetyl content in the starch.

The oil absorption capacity (OAC) of the starch samples also ranged between 57.8 and 98.2% with samples ASPS and UNSPS giving the lowest and highest values, respectively. Acetylation led to a general decrease in the OAC of the modified starches when compared to the unmodified native starches (UNSPS and UNWYS). The reason that can be attributed to this observation has to do with lower value of acetyl content in the starch molecules which might have caused minimal oil binding capacity due to structural re-orientation of the starch molecules in



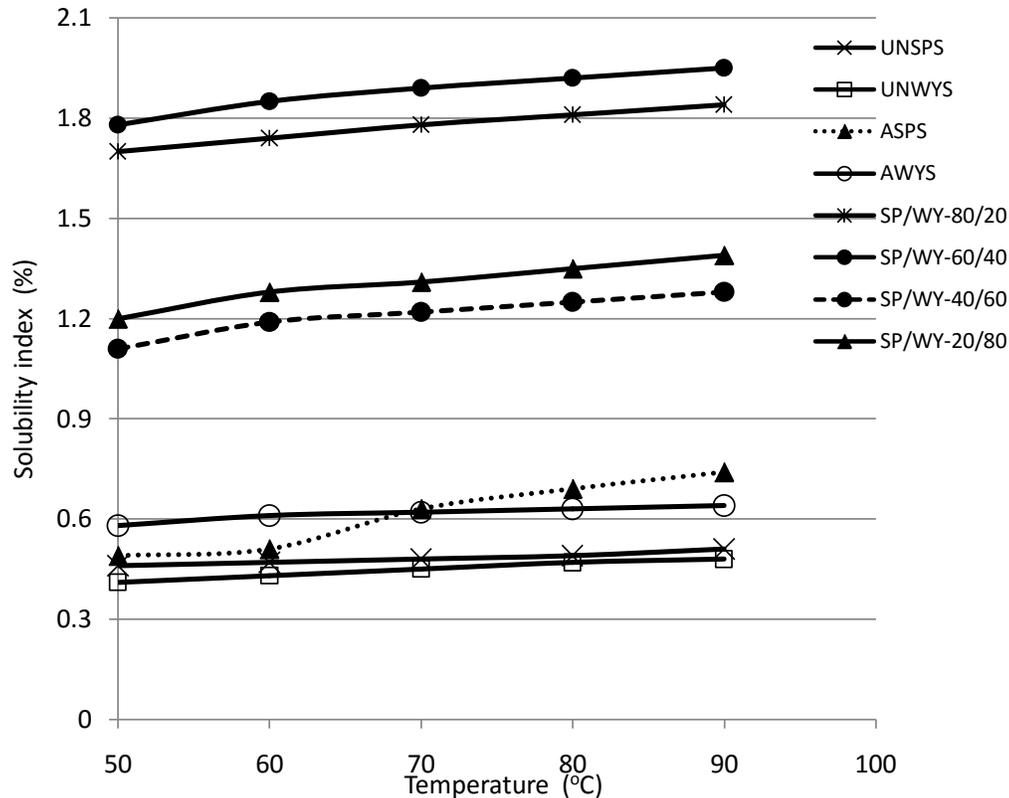
**Figure 2.** Swelling capacity of composited starches as influenced by acetylation and temperature. UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio.

comparison with the unmodified native starches (UNSPS and UNWYS). However, the observation in this study is contrary to some previous findings which showed that acetylation improved OAC in waxy maize starch (Biliaderis, 1982) and mucuna bean starch (Adebowale and Lawal, 2003). The degree of acetylation in the starch molecules might be responsible for this contradiction.

#### Effect of acetylation and temperature on the swelling capacity of composited starches

The swelling capacity of composited starches as influenced by acetylation and temperature is presented in Figure 2. There was generally an increase in the swelling capacity of all the starch samples with an increase in temperature from 50 to 90°C. For the unmodified starches, the swelling capacity of UNSPS at 50°C was 0.6% while that of UNWYS was 3.8%. However, at 90°C the value increased to 1.7% for UNSPS while that of UNWYS was 8.1%. The difference in the swelling capacity of the native starches may be attributed to the difference in the strength of associative binding forces within the starch granules (BeMiller and Whistler, 1996). The combined effect of starch compositing, acetylation and elevated temperature on the swelling capacity of the

starches was observed to be synergistic and counteracting in nature. At 50°C, the swelling capacity values of AWYS and SP/WY-20/80 were 9.8 and 10.4%, respectively. At 90°C, the value of AWYS rose to 12.8% while that of SP/WY-20/80 was 12.6%. Sample AWYS exhibited higher swelling capacity than their native starch counterparts which is a reflection of synergistic influence of acetylation and elevated temperature on the swelling capacity. In the case of SP/WY-20/80, higher swelling capacity exhibited was attributed to the synergistic influence of compositing, acetylation and elevated temperature on the swelling capacity. However, the swelling capacity values of ASPS, SP/WY-80/20, SP/WY-60/40 and SP/WY-40/60 at 50°C were 2.5, 3.4, 2.1 and 0.7%, respectively while at 90°C, the values rose to 4.2, 6.2, 5.3 and 3.8%, respectively. These values fell between that of the two native starches (UNSPS and UNWYS) which seem to be a reflection of counteracting influence of starch compositing on the swelling capacity. A previous finding has indicated that the individualistic role of acetylation on starches is such that the acetyl groups introduced would cause structural re-organisation of starch molecules thereby facilitating increased water percolation within the granules with subsequent increase in the swelling capacity (Jarowenko, 1986). Similarly, the individualistic role of elevated temperature on hydrated



**Figure 3.** Solubility index of composited starches as influenced by acetylation and temperature. UNSPS = Unmodified native sweet potato starch; UNWYS = Unmodified native water yam starch; ASPS = Acetylated sweet potato starch; AWYS = Acetylated water yam starch; SP/WY-80/20 = Acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = Acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = Acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = Acetylated sweet potato/water yam starch blend at 20/80 ratio.

starches is such that the associative binding forces within the starch granules would be weakened thereby causing a progressive hydration and subsequent swelling (Pal et al., 2002; Peroni et al., 2006). The individualistic role of starch compositing on the swelling capacity, in the present study, has revealed a synergistic and counteracting influence which may be attributed to the botanical origin of the starches involved in such compositing.

#### Effect of acetylation and temperature on the solubility index of composited starches

The solubility index of each of the acetylated starch samples showed that the values were greater than that of the unmodified native starch from sweet potato and water yam, respectively (Figure 3). The solubility index was also increasing with an increase in temperature for all the starch samples. At 50°C, SP/WY-60/40 gave the highest value (1.78%) of solubility index while ASPS gave the lowest value (0.49%); among the acetylated samples.

Similarly at 90°C, the highest solubility index was from SP/WY-60/40 (1.95%) while the lowest value was from AWYS (0.64%); among the acetylated samples. The implication of this observation is that the combination of acetylation process and starch compositing also had a synergistic effect on the solubility index of starches from sweet potato/water yam blends. It has earlier been observed that at an elevated temperature, the intra-granular binding forces within a starch molecule usually becomes weakened, causing the motional freedom of starch chains thereby facilitating increased solubility of the starch (Lawal, 2011). Similarly, Betancur and Chel (1997) and Bello-Perez et al. (1999) postulated that when a starch is acetylated, the acetyl groups introduced into the starch molecules usually allow retention of water molecules and enable better dispersion of starch in aqueous systems. This was attributed to their ability to form hydrogen bonds and prevent chain association, hence an increased swelling capacity and solubility of the starch. Therefore, the synergistic influence of acetylation and starch compositing on the solubility index may be attributed to enhanced water molecule retention within

**Table 2.** Colour characteristics of composited starches as influenced by acetylation.

Starch sample <sup>1</sup>	Colour variable <sup>2</sup>			
	L*	a*	b*	Chroma (C)
UNSPS	92.18±0.09 <sup>d</sup>	1.33±0.06 <sup>a</sup>	10.17±0.27 <sup>d</sup>	10.26±0.28 <sup>c</sup>
UNWYS	92.31±0.13 <sup>d</sup>	1.27±0.04 <sup>a</sup>	10.62±0.49 <sup>d</sup>	10.71±0.49 <sup>c</sup>
ASPS	94.02±0.12 <sup>a</sup>	0.75±0.03 <sup>e</sup>	13.34±0.46 <sup>a</sup>	13.36±0.46 <sup>a</sup>
AWYS	93.31±0.15 <sup>b</sup>	0.92±0.04 <sup>d</sup>	12.24±0.77 <sup>bc</sup>	12.27±0.77 <sup>b</sup>
SP/WY-80/20	93.06±0.09 <sup>c</sup>	1.04±0.05 <sup>c</sup>	11.79±0.49 <sup>bc</sup>	11.84±0.49 <sup>b</sup>
SP/WY-60/40	93.05±0.11 <sup>c</sup>	1.12±0.03 <sup>b</sup>	11.71±0.55 <sup>c</sup>	11.76±0.55 <sup>b</sup>
SP/WY-40/60	93.06±0.08 <sup>c</sup>	1.04±0.04 <sup>c</sup>	11.79±0.31 <sup>bc</sup>	11.84±0.31 <sup>b</sup>
SP/WY-20/80	93.91±0.14 <sup>a</sup>	0.87±0.02 <sup>d</sup>	12.67±0.42 <sup>ab</sup>	12.71±0.42 <sup>ab</sup>

<sup>1</sup>Starch sample: UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio. <sup>2</sup>Mean values followed by different superscripts in the column are significantly different at  $p < 0.05$ .

starch molecules and starch dispersion in aqueous systems. Some previous findings had also revealed that acetylation has the capacity to increase the swelling power and solubility of modified starches (Sodhi and Singh, 2012; Ali and Hasnain, 2014).

### Colour characteristics of composited starches as influenced by acetylation

The lightness index (L\*-value) of all the acetylated starch samples indicated that the values were higher than that of the unmodified native starches from sweet potato and water yam, respectively (Table 2). Sample ASPS gave the highest L\*-value (94.02) while SP/WY-60/40 gave the lowest L\*-value (93.05); among the acetylated starches. The implication of this observation is that acetylation seems to have a brightening effect on the starch samples. It has earlier been reported that acetylation has the capacity to improve the appearance of starch during processing due to its ability to prevent possible chemical reactions that may cause discolouration (Jarowenko, 1986; Satin, 1998).

The chroma, C-value, of the starch samples also increased in the acetylated samples as against the unmodified native starches (UNSPS and UNWYS). The C-values for SP/WY-80/20, SP/WY-60/40, SP/WY-40/60 and SP/WY-20/80 were 11.84, 11.76, 11.84 and 12.71, respectively while those of UNSPS and UNWYS were 10.26 and 10.71, respectively. The chroma has been reported to be a measure of colour purity in a material (Deman, 1990). The '±a\*' is regarded as a measure of the degree of redness or greenness while '±b\*' represents the degree of yellowness or blueness in a material (Giese, 2000). However, these factors may not be useful indices for describing the colour characteristics of starches although their popular use lies in the visual

colour assessment in fruit ripening (Ferrer et al., 2005).

### Effect of acetylation on the pasting properties of composited starches

The pasting properties of composited starches as influenced by acetylation are presented in Table 3. The peak viscosities of all the acetylated starches (composited and non-composited) were lower than that of the unmodified native starches (UNSPS and UNWYS). The peak viscosity ranged between 292.4 and 652 RVU as against the higher values of UNSPS (719.9 RVU) and UNWYS (743.2 RVU). Certain factors have been identified to influence the peak viscosity of acetylated starches and these include the botanical sources of the starch and the type of reagents involved in the acetylation process (Wilkins et al., 2003). A similar decrease in peak viscosity was also observed for acetylated maize starch (Nunez-Santiago et al., 2011) and white sorghum starch (Ali and Hasnain, 2014). However, certain workers have shown a higher peak viscosity in acetylated starches than that of their native starch counterparts and these include banana starch (Reddy et al., 2014), commercial corn hybrid starch (Wilkins et al., 2003) and starch from acha grains (Olu-Owolabi et al., 2014). The significant differences in the peak viscosity values of the starches, in this study, may be attributed to differences in the rate of water absorption by the starches as well as in the rate of starch granule swelling during heating (Ragaei and Abdel-Aal, 2006).

The breakdown viscosity of the acetylated starches (composited and non-composited) ranged from 164.1 to 323.3 RVU as against the higher values for the unmodified starches (UNSPS and UNWYS). A higher breakdown viscosity connotes relative paste instability during cooking while a lower value indicates relative

**Table 3.** Pasting properties of composited starches as influenced by acetylation.

Starch sample <sup>1</sup>	Pasting variable <sup>2</sup>						
	Peak viscosity (RVU)	Trough (RVU)	Breakdown viscosity (RVU)	Final viscosity (RVU)	Setback viscosity (RVU)	Peak temperature (°C)	Peak time (min)
UNSPS	719.9±7.8 <sup>b</sup>	345.9±2.5 <sup>c</sup>	374.0±3.2 <sup>b</sup>	541.7±3.7 <sup>b</sup>	195.8±2.1 <sup>a</sup>	80.3±0.2 <sup>c</sup>	4.2±0.1 <sup>b</sup>
UNWYS	743.2±8.6 <sup>a</sup>	360.4±3.1 <sup>b</sup>	382.8±3.5 <sup>a</sup>	558.3±3.1 <sup>a</sup>	197.9±1.4 <sup>a</sup>	81.6±0.2 <sup>a</sup>	4.2±0.1 <sup>b</sup>
ASPS	292.4±4.2 <sup>f</sup>	105.1±1.6 <sup>h</sup>	187.7±2.6 <sup>e</sup>	175.0±1.8 <sup>h</sup>	69.9±1.5 <sup>e</sup>	81.5±0.2 <sup>a</sup>	4.6±0.1 <sup>a</sup>
AWYS	426.5±3.3 <sup>d</sup>	242.8±2.2 <sup>e</sup>	183.7±1.9 <sup>e</sup>	437.5±2.8 <sup>e</sup>	194.7±2.8 <sup>a</sup>	81.5±0.1 <sup>a</sup>	4.6±0.1 <sup>a</sup>
SP/WY-80/20	292.8±4.1 <sup>f</sup>	128.7±1.4 <sup>g</sup>	164.1±2.1 <sup>f</sup>	191.7±1.6 <sup>g</sup>	63.0±1.1 <sup>f</sup>	81.5±0.2 <sup>a</sup>	4.2±0.1 <sup>b</sup>
SP/WY-60/40	651.9±5.9 <sup>c</sup>	328.6±2.8 <sup>d</sup>	323.3±2.6 <sup>c</sup>	458.3±3.9 <sup>d</sup>	129.7±1.4 <sup>d</sup>	80.4±0.1 <sup>c</sup>	4.2±0.1 <sup>b</sup>
SP/WY-40/60	652.0±9.2 <sup>c</sup>	377.3±3.9 <sup>a</sup>	274.7±2.2 <sup>d</sup>	525.0±4.1 <sup>c</sup>	147.7±1.8 <sup>c</sup>	80.7±0.1 <sup>b</sup>	4.2±0.1 <sup>b</sup>
SP/WY-20/80	401.8±4.6 <sup>e</sup>	236.9±1.9 <sup>f</sup>	164.9±1.8 <sup>f</sup>	425.0±2.6 <sup>f</sup>	188.1±1.7 <sup>b</sup>	80.7±0.1 <sup>b</sup>	4.6±0.1 <sup>a</sup>

<sup>1</sup>Starch sample: UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio. <sup>2</sup>Mean values followed by different superscripts in the same column are significantly different at  $p < 0.05$ .

paste stability (Newport-Scientific, 1996). Some workers have similarly discovered a lower breakdown viscosity in acetylated starches than that of the native starches. These include the acetylated potato starch (Nunez-Santiago et al., 2011) and acetylated acha starch (Olu-Owolabi et al., 2014). However, a contrary observation was made in the case of acetylated banana starch which exhibited a higher breakdown viscosity than that of the native starch (Reddy et al., 2014).

The final viscosity of the acetylated starches (composited and non-composited) ranged between 175 and 525 RVU, lower than that of the unmodified starches of UNSPS (541.7 RVU) and UNWYS (558.3 RVU). For the composited starches, the final viscosity values of SP/WY-80/20, SP/WY-60/40, SP/WY-40/60 and SP/WY-20/80 were 191.7, 458.3, 525 and 425 RVU, respectively. Some previous findings that similarly showed lower final viscosity in acetylated starches than that of the native starches include acetylated maize starch (Nunez-Santiago et al., 2011) and acetylated banana starch (Reddy et al., 2014).

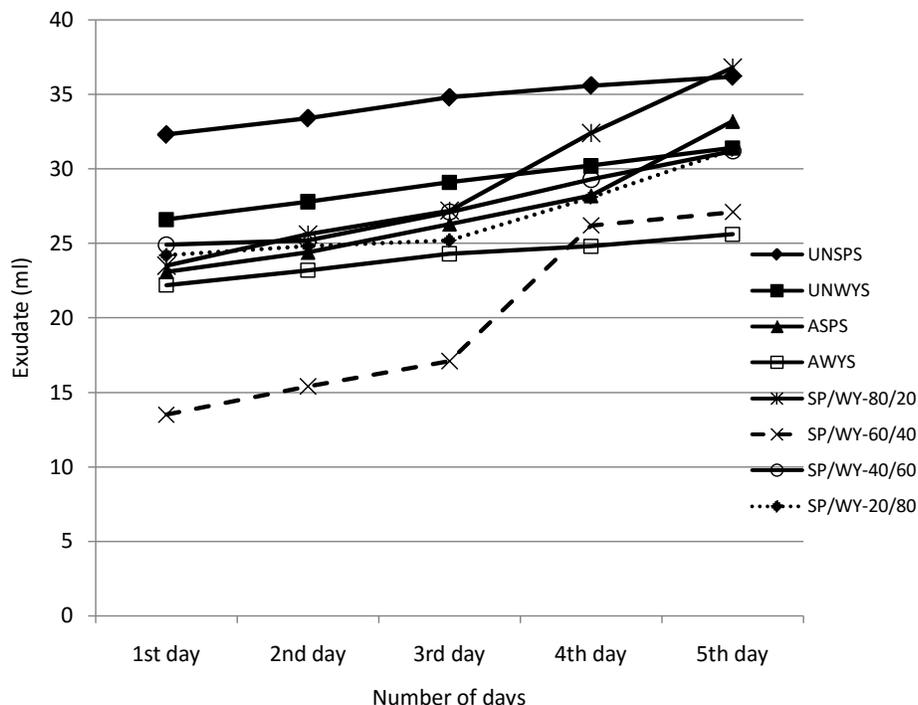
The setback viscosity values of the native starches were 195.8 RVU (UNSPS) and 197.9 RVU (UNWYS); higher than that of the acetylated starches (composited and non-composited) which ranged between 63 and 188.1 RVU. The setback viscosity has been observed to be an indicator for measuring the extent of retrogradation tendency or re-alignment process in the starch molecules particularly during cooling (Sandhu and Singh, 2007). Therefore, the starch samples with relatively high setback viscosity would, most probably, exhibit a higher retrogradation tendency (Bolade and Adeyemi, 2012). Some previous findings that similarly indicated a lower setback viscosity in acetylated starches than that of the native starches include that of acetylated acha starch (Olu-Owolabi et al., 2014), acetylated banana starch

(Reddy et al., 2014) and acetylated sorghum starch (Sodhi and Singh, 2012).

The peak temperature of the acetylated starches was found to be between that of UNSPS (80.3°C) and UNWYS (81.6°C). Similarly, the peak time of the acetylated starches ranged between 4.2 and 4.6 min while that of the native starches remained at 4.2 min for both UNSPS and UNWYS. The general observation in this study was that acetylation seemed to have an enhancing power on both the peak temperature and time while starch compositing itself had a counteracting influence on both parameters. However, a contrary observation was made in the case of white sorghum starch where acetylation led to a lower peak temperature and time than the unmodified counterpart (Ali and Hasnain, 2014).

#### Freeze-thaw stability of gels from composited starches as influenced by acetylation

The freeze-thaw stability of gels from the composited starches as influenced by acetylation is presented in Figure 4. The gels from acetylated starches generally had lower exudate than that of the native starches ranging between 13.5 ml (SP/WY-60/40) and 23.5 ml (SP/WY-40/60); at 1<sup>st</sup> freeze-thaw cycle. Higher exudates obtained from the native starches also at 1<sup>st</sup> freeze-thaw cycle were 32.3 ml (UNSPS) and 26.6 ml (UNWYS). It was observed that the exudate level in the gels was increasing with an increase in the number of days (freeze-thaw cycles) for all the starch samples (composited and non-composited). At 5<sup>th</sup> freeze-thaw cycle, the exudate level of UNSPS increased to 36.2 ml while that of UNWYS increased to 31.4 ml. However, the exudate level of gels from the acetylated starches reduced to between 25.6 ml



**Figure 4.** Freeze-thaw stability of gels from composited starches as influenced by acetylation. UNSPS = Unmodified native sweet potato starch; UNWYS = unmodified native water yam starch; ASPS = Acetylated sweet potato starch; AWYS = acetylated water yam starch; SP/WY-80/20 = acetylated sweet potato/water yam starch blend at 80/20 ratio; SP/WY-60/40 = acetylated sweet potato/water yam starch blend at 60/40 ratio; SP/WY-40/60 = acetylated sweet potato/water yam starch blend at 40/60 ratio; SP/WY-20/80 = acetylated sweet potato/water yam starch blend at 20/80 ratio.

(SP/WY-60/40) and 36.8 ml (SP/WY-80/20); also at 5<sup>th</sup> freeze-thaw cycle. The lower exudate level in gels from the acetylated starches connotes higher freeze-thaw stability as well as reduced syneresis tendency particularly at very low temperature. It has earlier been observed that syneresis in freeze-thawed gels obtained from native starches is as a result of re-arrangement of amylose molecules in starch granules at low temperature which acts to exclude water from the gel structure (Ayucitra, 2012). However, in gels from acetylated starches, the acetyl groups present facilitate the prevention of the re-alignment and association between macromolecules thereby leading to reduced syneresis (Zheng and Sosulski, 1998; Sodhi and Singh, 2012).

## Conclusion

The acetylation of composited starches from sweet potato and water yam has revealed that the physicochemical properties of the modified starches could be greatly influenced. The water absorption capacity of the acetylated starches was enhanced while the oil absorption capacity got reduced. The swelling capacity and colour characteristics of the acetylated starches revealed a

synergistic influence of acetylated and compositing as well as the counteracting role of compositing itself, in certain cases. The solubility indices of the acetylated starches were generally enhanced by acetylation and compositing. The pasting variables (peak, breakdown, final and setback viscosities) of the acetylated starches were all observed to be lower than that of the native starches. Acetylation enhanced the peak temperature and time of the modified starches while compositing however played a counteracting role. The freeze-thaw stability of gels from the acetylated starches was greatly improved as lower exudate was generated from the acetylated samples.

## Conflict of Interests

The authors have not declared any conflict of interests.

## REFERENCES

- Adebowale KO, Lawal OS (2003). Functional properties and retrogradation behaviour of native and chemically modified starch of mucuna bean (*Mucuna pruriens*). *J. Sci. Food Agric.* 83(15):1541-1546.
- Ali TM, Hasnain A (2014). Morphological, physicochemical, and pasting

- properties of modified white sorghum (*Sorghum bicolor*) starch. *Int. J. Food Prop.* 17(3):523-535.
- Ayucitra A (2012). Preparation and characterisation of acetylated corn starches. *Int. J. Chem. Eng. Appl.* 3(3):156-159.
- Bello-Pérez LA, Agama-Acevedo E, Sanchez-Hernandez L, Paredes-López O (1999). Isolation and partial characterization of banana starches. *J. Agric. Food Chem.* 47:854-857.
- BeMiller JN, Whistler RL (1996). Carbohydrates. In: *Food chemistry*, 3<sup>rd</sup> ed. Marcel Dekker Inc.: USA, pp. 157-223.
- Betancur AD, Chel GL (1997). Acid hydrolysis and characterization of *Canavalia ensiformis* starch. *J. Agric. Food Chem.* 45(11):4237-4241.
- Biliaderis CG (1982). Physical characteristics, enzymatic digestibility and structure of chemically-modified smooth pea and waxy maize starches. *J. Agric. Food Chem.* 30:925-930.
- Bolade MK, Adeyemi IA (2012). Functionality enhancement of composite cassava flour in the production of maize tuwo (a non-fermented maize-based food dumpling). *Food Bioprocess Technol.* 5:1340-1348.
- Bolade MK, Bello SB (2006). Selected physicochemical properties of flour from the root of African Fan Palm (*Borassus aethiopum*). *Int. J. Food Prop.* 9:701-713.
- Coursey DG, Ferber CEM (1979). The processing of yams. In: *Small-scale processing and storage of tropical root crops*. Westview Press: Colorado, pp. 189-212.
- Cousidine DM (1982). *Foods and food production encyclopedia*. John Wiley Inc.: New York, USA.
- DeMan JM (1990). *Principles of food chemistry*, 2<sup>nd</sup> ed. Van Nostrand Reinhold: New York, USA.
- Ferrer A, Remon S, Negueruela AI, Oria R (2005). Changes during the ripening of the very late season Spanish peach cultivar Calanda: Feasibility of using CIELAB coordinates as maturity indices. *Sci. Hortic.* 105:435-446.
- Giese J (2000). Colour measurement in foods as a quality parameter. *Food Technol.* 54(2):62-63.
- Golachowski A (2003). Properties of acetylated starch obtained from SO<sub>2</sub>-treated starch milk. *EJPAU- Food Sci. Technol.* 6(1):1-8.
- González Z, Perez E (2002). Effect of acetylation on some properties of rice starch. *Starch/Stärke* 54:148-154.
- Hermansson AM, Svegmarm K (1996). Developments in the understanding of starch functionality. *Trend Food Sci. Technol.* 7:345-353.
- Izuagie T, Hassan LG, Uba A, Achor M, Sahabi DM (2012). Composition and physicochemical properties of starch from christ thorn seeds. *Bayero J. Pure Appl. Sci.* 5(1):60-65.
- Jarowenko W (1986). Acetylated starch and miscellaneous organic esters. In: *Modified starches: properties and uses*. CRC Press: Boca Raton, FL, pp. 55-77.
- Kaur L, Singh N, Singh J (2004). Factors influencing the properties of hydroxypropylated potato starches. *Carbohydr. Polym.* 55(2):211-223.
- Lawal OS (2004). Composition, physicochemical properties and retrogradation characteristics of native, oxidised, acetylated and acid-thinned new cocoyam (*Xanthosoma sagittifolium*) starch. *Food Chem.* 87(2):205-218.
- Lawal OS (2011). Hydroxypropylation of pigeon pea (*Cajanus cajan*) starch: preparation, functional characterizations and enzymatic digestibility. *LWT Food Sci. Technol.* 44(3):771-778.
- Leach HW, McCowen LD, Schoch TJ (1959). Structure of the starch granule: swelling and solubility patterns of various starches. *Cereal Chem.* 36:534-544.
- McGuire RG (1992). Reporting of objective color measurement. *HortScience* 27:1254-1255.
- Mirmoghtadaie L, Kadivar M, Shahedi M (2009). Effects of cross-linking and acetylation on oat starch properties. *Food Chem.* 116:709-713.
- Newport-Scientific (1996). *Operational Manual for the Series 4 Rapid Visco Analyser*. Newport Scientific Pty, Ltd, Australia.
- Nunez-Santiago MC, Garcia-Suarez FJ, Gutierrez-Meraz F, Sanchez-Rivera MM, Bello-Perez LA (2011). Some intrinsic and extrinsic factors of acetylated starches: Morphological, physicochemical and structural characteristics. *Rev. Mex. Ing. Quim.* 10(3):501-512.
- Olu-Owolabi BI, Olayinka OO, Adegbemile AA, Adebowale KO (2014). Comparison of functional properties between native and chemically-modified starches from acha (*Digitaria staph*) grains. *Food Nutr. Sci.* 5:222-230.
- Pal J, Singhal RS, Kulkarni PR (2002). Physicochemical properties of hydroxypropyl derivative from corn and amaranth starch. *Carbohydr. Polym.* 48:49-53.
- Peroni FHG, Rocha TS, Franco CML (2006). Some structural and physicochemical characteristics of tuber and root starches. *Food Sci. Tech. Int.* 2(6):505-513.
- Ptaszek P, Grzesik M (2007). Viscoelastic properties of maize starch and guar gum gels. *J. Food Eng.* 82:227-237.
- Ragaei S, Abdel-Aal EM (2006). Pasting properties of starch and protein in selected cereals and quality of their food products. *Food Chem.* 95:9-18.
- Reddy CK, Haripriya S, Suriya M (2014). Effect of acetylation on morphology, pasting and functional properties of starch from banana (Musa AAB). *Ind. J. Sci. Res. Technol.* 2(6):31-36.
- Sandhu KS, Singh N (2007). Some properties of corn starches II: Physicochemical, gelatinization, retrogradation, pasting and gel textural properties. *Food Chem.* 101:1499-1507.
- Sathe SK, Deshpande SS, Salunkhe DK (1982). Functional properties of winged bean [*Psophocarpus tetragonolobus* (L.) DC] protein. *J. Food Sci.* 47:503-509.
- Sathe SK, Salunkhe DK (1981). Isolation, partial characterization and modification of the great Northern bean (*Phaseolus vulgaris* L.) starch. *J. Food Sci.* 46(2):617-621.
- Satin M (1998). *Functional properties of starches*. FAO report.
- Singh J, Kaur L, McCarthy OJ (2007). Factors influencing the physicochemical, morphological, thermal and rheological properties of some chemically modified starches for food applications - A review. *Food Hydrocoll.* 21:1-22.
- Sodhi NS, Singh N (2012). Structure and functional properties of acetylated sorghum starch. *Int. J. Food Prop.* 15(2):312-325.
- Tewe OO, Ojeniyi FE, Abu OA (2003). Sweet potato production, utilization and marketing in Nigeria. International Potato Center (CIP): Peru.
- Thomas DJ, Atwell WA (1997). *Starches: Practical guides for the food industries*. Eagan Press: USA.
- Wilkins MR, Wang P, Xu L, Niu Y, Tumbleson ME, Rausch KD (2003). Variability of reaction efficiencies and pasting properties of acetylated dent corn starch from various commercial hybrids. *Cereal Chem.* 80(1):72-75.
- Xu Y, Miladinov V, Hanna MA (2004). Synthesis and characterization of starch acetates with high substitution. *Cereal Chem.* 81(6):735-740.
- Zheng GH, Sosulski FW (1998). Determination of water separation from cooked starch and flour pastes after refrigeration and freeze-thaw. *J. Food Sci.* 63(1):134-139.