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PHYSICOCHEMICAL PROPERTIES OF MODIFIED TRIFOLIATE YAM STARCHES

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ABSTRACT

Physicochemical properties of modified trifoliate yam starches were determined. Trifoliate yam starches were harvested and the starch was extracted. The extracted starch was modified using four methods (acid and alkali treatment, oxidation and acetylation). Colour and physicochemical properties of the modified starches were determined. Colour of pre-gelatinized trifoliate yam starch was significantly different ($p<0.05$) from other starches. The colour was yellowish/reddish while other starches were lighter in colour. The structures of untreated, oxidized, pre-gelatinized and acetylated starches were polygonal in shape. Granule sizes of native and modified trifoliate yam starches ranged from 2.53 to 3.86 µm. Amylose content of trifoliate yam starches ranged from 10.01 to 16.17%. Swelling power of pre-gelatinized starch increased from 2.15 at 60 °C to 3.00 at 70 °C but the values decreased at 80 to 90 °C. Acid-treated starch exhibited higher paste clarity at refrigerated temperature than ambient temperature but at ambient temperature, oxidized starch had high paste clarity during storage. Acid and alkali-treated starches had higher water exudate at day 0 which later decreased with storage. Acetylated starch had higher peak viscosity (3833.00 cP). Acid and alkali treatment reduced the viscosities and had low freeze-thaw stability.

Keywords: Colour, Modification, Physicochemical, Trifoliate yam starch

INTRODUCTION

Starch is one of the most important polysaccharides found in many food plants and consists of amylose and branched amylopectin molecules (Tako *et al.*, 2014). According to Santana *et al.* (2014), starch is a macro-constituent of many foods and is widely consumed by humans as an inexpensive and stable carbohydrate source. It is useful in food production, cosmetics, paper, textile, as adhesive, thickening, stabilizing, stiffening and gelling agents (Tako *et al.*, 2014). Thys *et al.* (2013) also reported starch as the most common thickening and gelling agent used by the food industry in the development of a large number of products such as soups, sauces, and ready-to-eat food among others.

Native starches have some limitations in their use in the food industry due to low shear resistance, thermal resistance, thermal decomposition and a high tendency towards retrogradation (Odeniyi and Ayorinde, 2012; Otegbayo *et al.* 2013). Therefore, starch can be structurally modified by various means to enhance its functions as an ingredient (Chung *et al.*, 2008; Eliasson and Gudmundsson, 1996). Native starches are modified in order to improve their functional properties and these can be done using physical (heat-moisture, steam-pressure, microwave irradiated), chemical (acid treatment, oxidation, cross-linking, acetylation, succinylation, phosphorylation, hydroxypropylation and carboxymethylation) and enzymatic technique (Abbas *et al.*, 2010; Jyothi *et al.*, 2013). Modification of other starches from corn, cassava, potato and some yam species had been reported (Jyothi *et al.*, 2013; Adetunji *et al.*, 2006; Zięba *et al.*, 2010; Okunola and Akingbala, 2013). Modification of trifoliate yam starches for industrial application had not been extensively researched like other yam species. This may be due to the problems (hardening and presence of bitter principle) associated with this yam species thereby limiting the cultivation and market value of the crop. Starch granules of trifoliate yam are smaller than other yam species which signified higher digestibility of the starch (Akinoso and Abiodun, 2013). Therefore, this work determined the physicochemical properties of modified trifoliate yam starches.

MATERIALS AND METHODS

Freshly harvested trifoliate yam tubers were obtained at a farm in Osun State Polytechnic, Iree, Nigeria. Method of Akinwande *et al.*, (2007) was used for starch extraction. Acid-modified starch was produced according to Okunola and Akingbala (2013) using 6% HCl solution and neutralized with 10% (w/v) NaOH solution. Alkali-modified starch was produced using the method of Gutiérrez *et al.,* (2014) while acetylated and oxidized starches were produced using methods of Sathe and Salunkhe (1981) and Iheagwara (2012) respectively. Pre-gelatinized starch was done according to method of Oladebeye *et al.*, (2011).

The colour attributes (L, a, and b values) of the yam starch was measured using a Minolta portable chroma-metre. The colour coordinates system L* a* and b* values were recorded.

Morphological structure of starch was done using a light microscope (LM) and stained with drops of potassium iodide (KI) solution. The images were viewed at a magnification of x 400 (AmScope Binocular Digital Microscope version x86, 3.7.4183). The area, perimeter and diameter of the starch granules were measured and used to calculate the granule sizes and form factor. Amylose determination was done using the method of Juliano (1971). The stability and clarity of starch pastes were determined at 27 °C and 4 °C following the method of Gutiérrez *et al.*, (2014). The percentage (%) transmittance at 650 nm was determined in a spectrophotometer (BK-UV1600PC) using distilled water as a blank. Method of Lutfi and Hasnain (2013) was used for freeze-thaw stability at 5% starch solution.

Moisture and swelling power determination were carried out using the procedure of AOAC method 934.01 (AOAC, 2006) and Peroni *et al.,* (2006) respectively. The pasting profile of the starch sample was studied using a Rapid Visco-Analyzer (RVA) (Newport Scientific Pty Ltd) with the aid of a thermocline for windows version 1.1 software.

Statistical Analysis

All procedures were carried out in triplicates. The mean and standard deviation of the data obtained was calculated. The data were evaluated for significant differences in their means using Analysis of Variance at $p \leq 0.05$. Differences between the means were separated using Tukey's test.

RESULTS AND DISCUSSION

Effect of modification on colour of trifoliate yam starches

Alkali-treated starch was significantly different ($p<0.05$) from oxidized and pre-gelatinized starches in lightness value (Table I). The alkali-treated starch colour was lighter than other starches but the value obtained was not significantly different ($p<0.05$) from native, acetylated and acid-treated starches. Modification significantly improved the color value (whiteness) of acid and alkali-treated, acetylated and oxidized starches. Pre-gelatinized starch had lower L* value (59.66) but higher a* and b* values of 3.76 and 9.40 respectively, which were significantly different ($p<0.05$) from other starches. Pre-gelatinized starch colour tends toward reddish and yellowish than other samples. Pre-gelatinized trifoliate yam starch could be used in products requiring coloured materials such as soup, jellies and so on due to imparted colour during modification. Colour is one of the important parameters that affect the product acceptability in the food industry.

Table I: Effect of modification on the colour of trifoliate yam starch

Treatment	L*	a*	b*
RY	87.99±1.11ab	1.29±0.06b	5.94±0.26c
AEY	86.46±0.87ab	0.88±0.14d	5.40±0.42c
ADY	88.100±0.39ab	1.12±0.07bc	7.31±0.07b
AKY	89.39±0.41a	0.96±0.08cd	7.63±0.12b
OY	86.09±1.66b	0.01±0.01e	6.02±0.51c
PY	59.66±1.79c	3.76±0.9a	9.40±0.23a

Values with the same superscript down the column were not significantly different ($p < 0.05$)
RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch

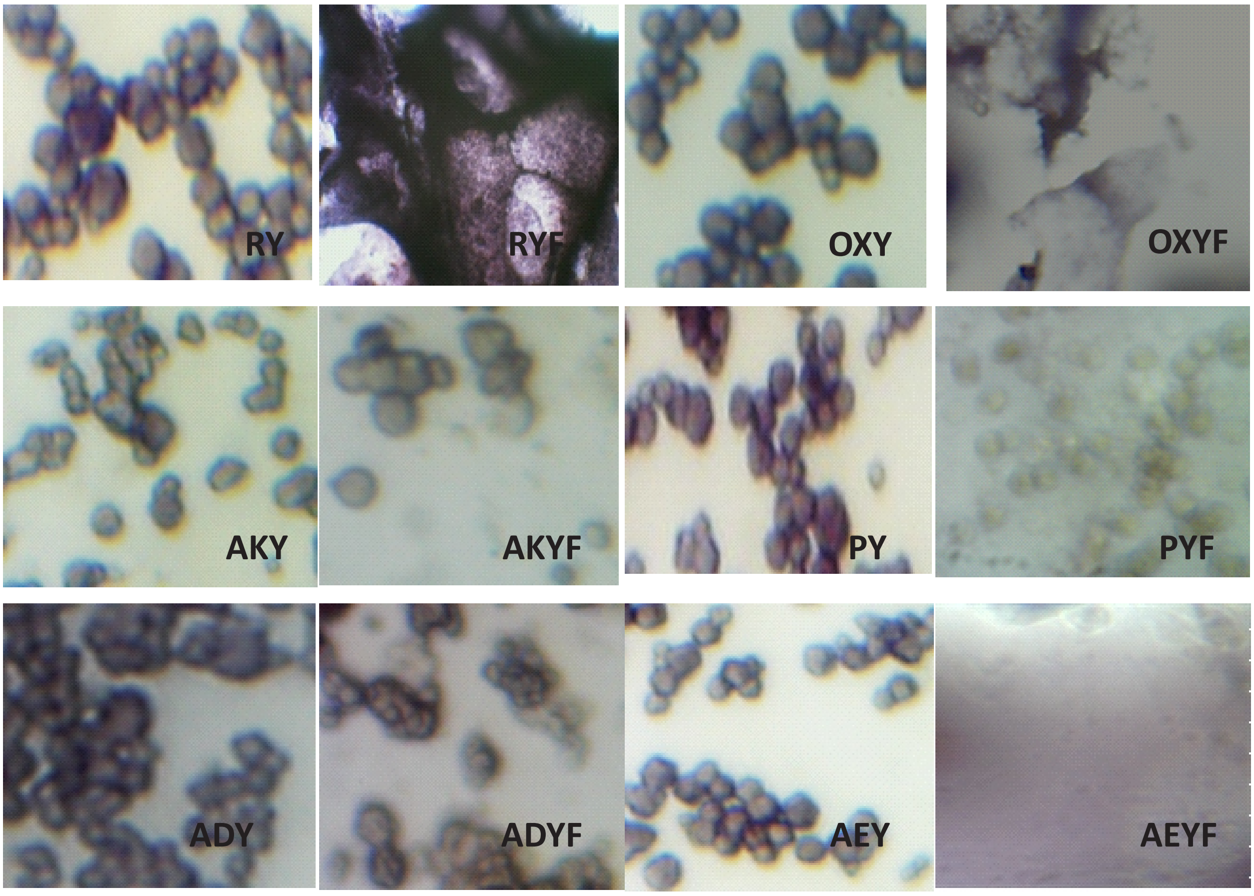

Figure I: Effect of modification and freeze-thaw on the morphological structure using light microscope (x 400 μm) of trifoliate yam starches

RY-Raw yam starch, RYF-Raw yam starch after freezing, AKY-Alkali-treated starch, AKYF-Alkali-treated starch after freezing, ADY-Acid-treated starch, ADYF-Acid-treated starch after freezing, OXY-Oxidized starch, OXYF-Oxidized starch after freezing, PY-Pre-gelatinized starch, PYF-Pre-gelatinized starch after freezing, AEY-Acetylated starch, AEYF-Acetylated starch after freezing

Effect of Modification on Morphological Structure of Trifoliate Yam Starches

The morphology of the starch granules is presented in figure I. The structures of untreated, oxidized, pre-gelatinized and acetylated starches were similar in shape. The shapes were polygonal but those of alkali and acid-treated starches did not have a definite shape and were attached. Acid and alkaline starches were not smooth like other untreated and other modified starches. After 10 freeze-thaw cycle, the untreated starch had a swollen starch structure with rough surfaces. Alkali and acid-treated starches had rough surface granules joined together. The oxidized starch structure had smooth surfaces but with fissures while acetylated starch had rough surfaces with perforations. Sukhija *et al.*, (2016) observed surface fissures in case of oxidized and oxidized cross-linked starch and smooth surface of native, cross-linked and cross-linked oxidized starches. According to Wang and Copeland (2015), acid hydrolysis can significantly change the structural and functional properties of starch without disrupting its granular morphology and that amorphous regions are hydrolyzed to enhance the crystallinity and double helical content of acid-hydrolyzed starch.

Effect of Modification on the Granule Size, Amylose and Moisture Contents of Trifoliate Yam Starches

Granule sizes of trifoliate yam starches ranged from 2.53 to 3.86 μm (Table II). There were significant differences ($p > 0.05$) in the values obtained for pre-gelatinized starch (PY) and other starches. PY had the highest value in granule size (3.86 μm) while the least value was in acetylated starch (AEY). There were no significant differences ($p < 0.05$) in granule sizes of alkali and acid-treated starches. Starch granule size values were within the range reported by Akinoso and Abiodun (2013). Granule sizes of trifoliate yam starch are smaller and thereby are more digestible than some yam starches. Amylose content of trifoliate yam starches ranged from 10.01 to 16.17%. Amylose contents of acetylated starch were significantly different ($p>0.05$) from untreated and other modified starches. Oxidized starch had the lowest amylose content (10.01%) and was significantly different ($p > 0.05$) from other starches. Amylose contents of the native and modified starches were low when compared to other roots and tubers such as white yam *Dioscorea rotundata* species (27.48-31.55%), taro (27.65-35.90%), sweet potato (28.69%) (Addy *et al.*, 2014; Himeda *et al.*, 2012; Aprianita *et al.*, 2009). Low contents of amylose observed in the starches could enhance swelling of the starches when incorporated into food products (Tester and Morrison, 1990) with a low tendency to retrograde. Moisture content of alkali-modified starch was higher and significantly different ($p > 0.05$) from other starches. Moisture contents of acid, alkali and oxidized starches were higher than the recommended value for flour. Therefore, they could easily be attacked by microorganisms. Low moisture contents are required in order to extend the shelf life during storage. Untreated, acetylated and pre-gelatinized starches could keep longer than other modified starches due to their low moisture contents. However, acid, alkali and oxidized starches could be further dried to extend their keeping quality.

Table II: Effect of modification on the granule size, amylose and moisture values of trifoliate yam starches

Treatment	Granule size (μm)	Amylose (%)	Moisture Contents (%)
RY	3.50±1.23b	15.57±0.49b	13.50±0.45d
AEY	2.53±2.07e	16.17±0.63a	10.18±0.37f
ADY	2.65±1.89d	11.36±0.72d	17.04±0.43b
AKY	2.66±0.97d	10.20±0.88e	17.85±0.21a
OY	2.91±0.81c	10.01±0.39e	16.09±0.40c
PY	3.86±1.55a	14.05±0.64c	10.50±0.39e

Values with the same superscript down the column were not significantly different ($p < 0.05$)
RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch

Effect of Modification on the Swelling Power of Trifoliate Yam Starches

Data for the swelling power of the starches are shown in figure II. Pre-gelatinized starch increased from 2.15 at 60 °C to 3.00 at 70 °C. The values of pre-gelatinized starches decreased at 80 to 90 °C. This indicated that the pre-gelatinized starch swelled at low temperature from 60 to 70 °C and reduction in the values occurred at higher temperatures from 80 °C and above. This may be due to the treatment (cooking) given to the starch as this caused reduction in the swelling ability of the starch at high temperatures. Native, acetylated and oxidized starches had similar trend in swelling power. The starches increased with increase in temperature with acetylated having a higher value (2.47) than the native (2.33) and oxidized (2.01) starches at 90 °C. According to Bello-Pérez *et al.,* (2006) increase in swelling power is related to the breaking of intermolecular hydrogen bonds in amorphous areas allowing progressive water absorption. There was a reduction in the swelling power of acid and alkali modified starches at high temperatures. The values were higher at 60 °C but decline till 90 °C in both starches. This showed that acid and alkali- modified starches had low tendency to swell. The starch components dissolved in the medium and form a clear liquid solution. The higher the temperature, the more the starch dissolves in the medium thereby reducing the starch sediments. Acid and alkali treatment cause hydrolysis of the starch components thereby making the starch miscible with the medium. The swelling power of acid-thinned starches decreased with the increase temperature. Thys *et al.* (2013) and Sandhu *et al.*, (2007) explained that during acid hydrolysis the amylose chains are fragmented forming a disorganized structure that cannot retain water during temperature increases. Vaclavik and Christian (2008) also observed that hydrolysis of the starch molecule results in less water absorption by the starch granule, thus causing thinner hot paste and less firm cooled product.

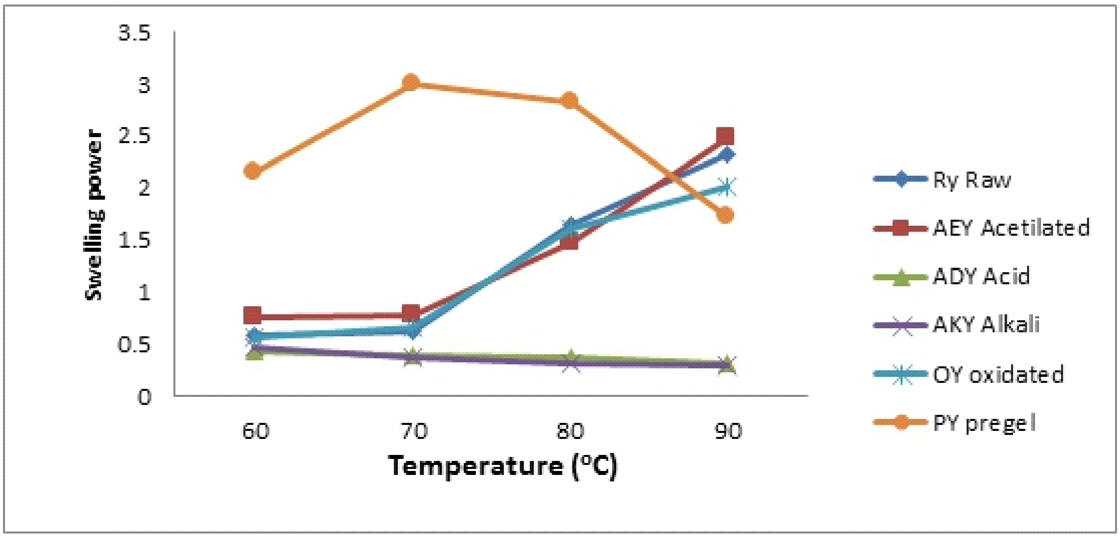

Figure II: Effect of modification on the swelling power of trifoliate yam starches
RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch

Effect of Modification on Paste Clarity of Trifoliate Yam Starches

The initial paste clarity was high in acid and alkali-modified starches with values of 22.9% and 20.9% respectively (Figure III). At 4 °C, the transmittance of acid-modified starch reduced (6.4%) drastically at 24 h interval but at 48 h interval, the transmittance increased slightly and later declined to 6.1%. For alkaline treated starch, transmittance also reduced to 2.7% at 24 h and there was a decrease in the values till 72 h. Other starches such as the untreated, acetylated, oxidized and pre-gelatinized starches decreased in clarity with storage days. For the samples stored at ambient temperature, a sharp increase was noticed with oxidized starch from 2.0% to 17.6% at 24 h and later declined till 72 h. For the acid and alkali-modified starches, rapid reduction in transmittance was noticed at 24 h but there was an increase in the values at 48 and 72 h. The same trend was observed in the untreated starch and pre-gelatinized starches as there was a reduction in transmittance at 24 h while the values increased at 48 h but later declined at 72 h. Native and modified trifoliate yam starches were clearer after preparation of the solution but became opaque and cloudy with storage both at ambient and refrigerated temperatures. Acid-treated starch exhibited higher paste clarity at refrigerated temperature than ambient temperature but at ambient temperature, oxidized starch had higher paste clarity during storage at 24 and 48 h. Starch paste clarity decreased during storage and this was in agreement with the report of Haghayegh and Schoenlechner (2010). According to Craig *et al.*, (1989), low transmittance indicated high opacity in the starch solution.

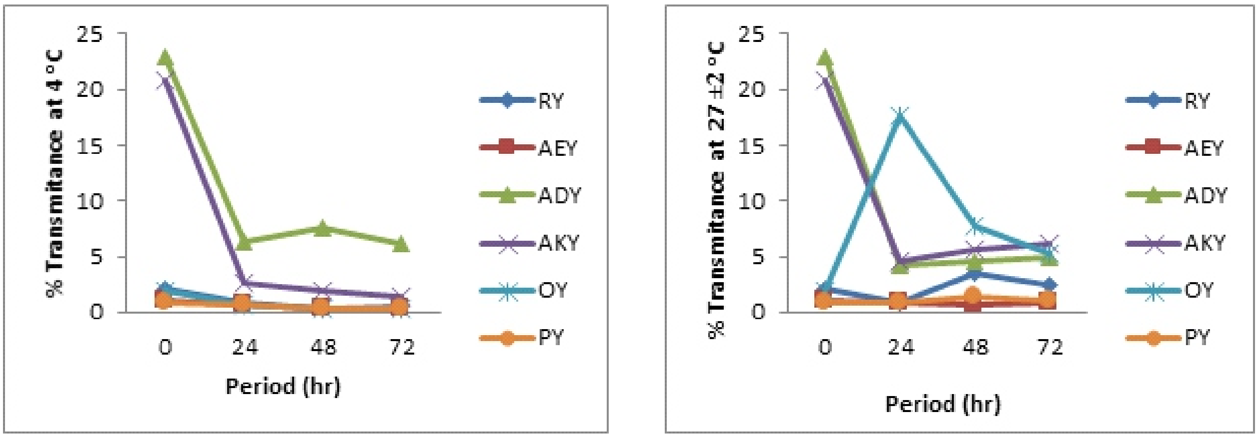

Figure III: Effect of modification on the percentage transmittance of trifoliate yam starches
RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch

Effect of Modification on Freeze-Thaw Stability of Trifoliate Yam Starches

Native and modified starches had higher water exudate at day 0 (Figure IV). The values decreased in all the starches at day 3 and there was a progressive increase in the amount of water exudate at day 10. Acetylated starch had higher freeze-thaw stability due to the least amount of water exudate observed in the starches during storage. The oxidized starch also had low freeze-thaw stability than the pre-gelatinized, acid and alkali starches. Acid and alkali-treated starches had low freeze-thaw stability throughout the storage periods. The amount of water exudate was due to the re-association of starch molecules, resulting in the formation of insoluble aggregates. However, Ferrero *et al.,* (1994) and Lutfi and Hasnain (2013) classified the gels as weepy, grainy, or spongy in nature. Furthermore, Ferrero *et al.,* (1994) explained further that during freezing, starch regions were created in the matrix and the water present in the matrix remained partially unfrozen. The concentration of starch in the matrix affects the association of starch chains in forming thick filament. However, the water molecules coagulate into ice crystals forming a separate phase in which the ice is converted to bulk phase water during thawing resulting in release of water from the polymeric network (syneresis). The water thus released leaves the starch gel with a sponge-like texture.

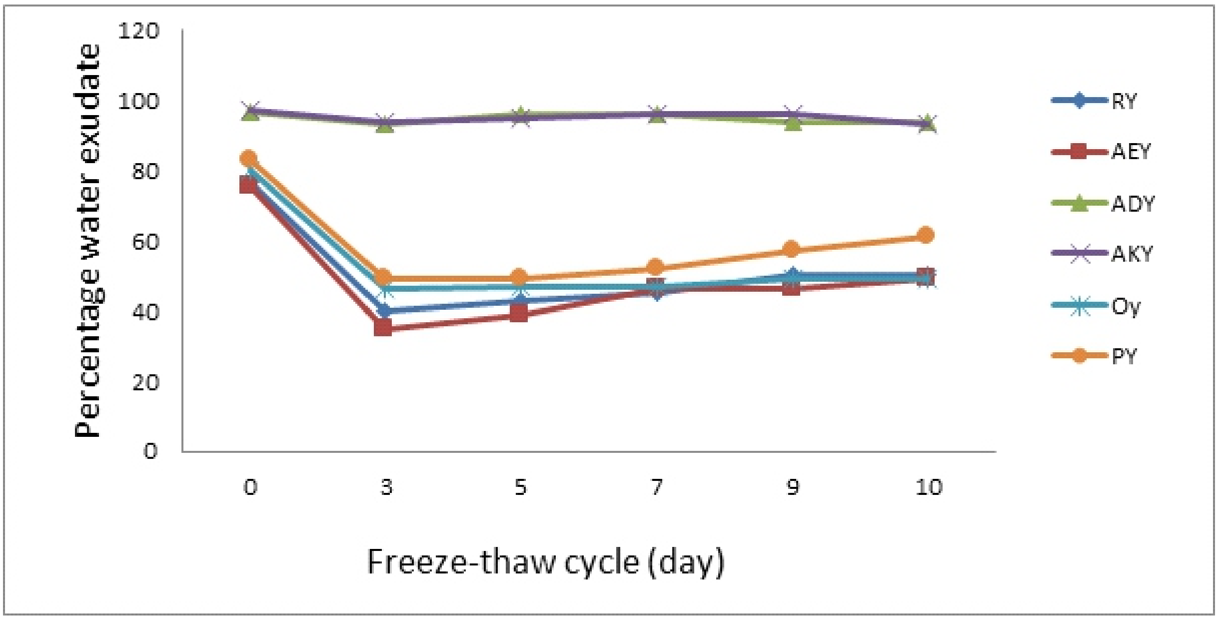

Figure IV: Effect of modification on freeze-thaw stability of trifoliate starches at 5% (w/v) Concentration

RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch

Effect of Modification on Pasting Properties of Trifoliate Yam Starches

Acetylated starch had higher peak viscosity (3833.00 cP) but was not significantly different ($p>0.05$) from untreated, oxidized and pre-gelatinized starches (Table III). No significant differences ($p>0.05$) were observed in the peak viscosity values of acid and alkali-treated starches. Pre-gelatinized starch had higher holding strength which was not significantly different ($p>0.05$) from the untreated starch sample. Untreated, acetylated and oxidized starches were not significantly different ($p>0.05$) from each other. Likewise, no significant differences ($p>0.05$) existed between the acid and alkali-treated starches in holding strength. Higher breakdown viscosity (2083.50 cP) was observed in acetylated starch followed by the untreated and oxidized starches respectively. No significant differences ($p < 0.05$) were observed in oxidized and pre-gelatinized starches, acid and alkali-treated starches in breakdown viscosities. Lower breakdown viscosities were observed in acid and alkali-treated starches with a value of 7.50 and 8.50 cP respectively. PY had higher final viscosity (5813.50 cP) and setback (3090.00 cP) respectively. These values were significantly different ($p < 0.05$) from the untreated and other modified starches. There were no significant differences ($p>0.05$) in the final viscosities and setback values obtained for untreated, acetylated and oxidized starches. However, the final viscosities, setback and pasting time values obtained for acid and alkali-treated starches were not significantly different ($p < 0.05$) from each other. PY had a higher cooking time (7.00 min) but at lower temperature (71.78 °C) than other starches. Pasting temperatures of untreated, acetylated and oxidized starches were not significantly different ($p < 0.05$) while the temperatures for acid and alkali-treated starches were not detected. Pasting temperature showed the minimum temperature for starch gelatinization. Moreover, Altay and Gunasekaran (2006) reported that starch gelatinization depends on water content, heating rate, the botanical source of starch, processes applied to starch before gelatinization, and amylose/amylopectin content of starch.

Table III: Effect of Modification on the Pasting Properties of Trifoliate Starches

Treatment	Peak (cP)	Holding strength (cP)	Breakdown (cP)	Final viscosity (cP)	Setback (cP)	Pasting time (min)	Pasting temperature (°C)
RY	3579.00 ±1.30a	2112.00±9.33ab	1467.00±3.68b	2797.00±7.50b	685.00±1.84b	5.10±0.04b	84.43±0.64a
AEY	3833.00±1.32a	1749.50±8.70b	2083.5±5.96a	2591.00±4.53b	841.50±4.17b	4.90±0.04c	83.28±0.04a
ADY	-29.00±2.83 b	-36.50±0.71c	7.50±2.12d	-33.50±0.71c	3.00±1.41c	1.10±0.04e	ND
AKY	-30.00±1.41b	-38.50±0.71c	8.50±0.71d	-36.00±0.00c	2.51±0.71c	1.07±0.00e	ND
OY	2964.50±1.95a	1915.5±1.04b	1049.00±9.50c	2581.50±1.10b	666.00±5.66b	4.67±0.09d	84.00±0.07a
PY	3628.50±5.34a	2723.00±3.68a	905.50±6.17c	5813.50±2.19a	3090.00±5.86a	7.00±0.00a	71.78±3.36b

Values with the same superscript down the column were not significantly different ($p < 0.05$)

RY- Raw yam starch, AKY-Alkali-treated starch, ADY-Acid-treated starch, OXY-Oxidized starch, PY-Pre-gelatinized starch, AEY-Acetylated starch, ND- Not detected

CONCLUSIONS

The work determined the effect of modification on the properties of trifoliate yam starch. The colour of alkali-treated starch was lighter than other starches. Pre-gelatinized starch was reddish/yellowish in colour and was significantly different ($p<0.05$) from other starches. Smaller granule sizes were observed for the starches. Acetylated starch had higher amylose content while the least value was observed in the oxidized starch. Pre-gelatinized, alkali and acid-treated starches swelled at lower temperature and decreased in swelling power at higher temperatures. However, oxidized, acetylated and native starches increased in swelling power with increased temperature. Decrease in paste clarity was observed for all the starches at 4 °C and at ambient temperature during storage. The amounts of water exudates in acid and alkali-treated starches were higher than other starches indicating low freeze-thaw stability. Peak viscosity was higher in acetylated starch while the native starch had higher final viscosity with higher setback value. Therefore, the starches could be used in different applications in food industry as thickeners, gel, stabilizer and in the food formulation.

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