ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

FT-IR Analysis and Modification Effects of Acetylation on Functional Properties of Starch extracted from Wheat (*Triticum aestivum* L.).

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ABSTRACT

Native wheat starch was isolated and subjected to chemical modifications using acetic anhydride to generate acetylated wheat starch. The native and the modified wheat starches were characterized in terms of their functional properties. The modification caused significant ($P<0.05$) difference in water absorption capacity, oil absorption capacity, swelling property, foaming capacity, gelation temperature and emulsion capacity. Fourier transform infrared spectroscopy (FTIR) results revealed a new band at 1240.23cm^{-1}

Keywords: *Triticum aestivum* L, gelatinization, functional groups, wheat starch

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ISSN: 0975-8585

INTRODUCTION

Starch is a natural polymer, principal storage polysaccharide of plants and made up with macromolecules of amylose (linear molecule) and amylopectin (branched). Starch contributes a fervent role in food processing industries in sustaining the quality characteristics including structure, thickening, gelling, consistency and shelf life stability to many food systems (Eliasson & Gudmundsson, 1996). Native starches, nevertheless of their sources are had less applications because of their incapability to tolerate processing conditions; the drawbacks are low paste clarity, sensitivity to pH, heat and shear, high retro gradation, low decomposition and syneresis, so there is a need to improve desirable functional properties. These drawbacks may overcome by modification of starches by physical (gamma irradiation and heat-moisture treatment), chemical (acetylation, acid-thinned, oxidation, cross-linking and esterification) and enzymatic process. In acetylation, the starch molecules are treated with specific amount of acetic anhydride under controlled affected factors of substrate concentration, reaction time, pH and structure of starches (Betancur-Ancona *et al*., 1997). In acetylation process, the hydrophilic hydroxyl groups are replaced with hydrophobic acetyl groups; it increases the hydrophobicity, prevents the hydrogen bonding formation between hydroxyl groups and water molecules. The acetylation process hindering or eliminating retro gradation will be affect steadying of the starch solution (Adebowale *et al*., 2002).

Wheat (*Triticum* spp.) is a cereal grain, originally from the Levant region of the Near East but now cultivated worldwide (Belderok *et al.,* 2000*)*. In 2013, world production of wheat was 713 million tons, making it the third most-produced cereal after maize 1,016 million tons and rice 745 million tons. Wheat was the second most-produced cereal in 2009; world production in that year was 682 million tons, after maize 817 million tons, and with rice as a close third 679 million tons (World Wheat, Corn and Rice 2015).

Globally, wheat is the leading source of vegetable protein in human food, having a higher protein content than other major cereals, maize (corn) or rice. In terms of total production tonnages used for food, it is currently second to rice as the main human food crop and ahead of maize, after allowing for maize's more extensive use in animal feeds. Due to high amount of starch, wheat processing into flour and starch is of interest in sight of a possibly significant resource for food and non-food industries. Further limited studies are available on modification of wheat starch and applicability of wheat starch in food and non-food industries. With this limited information the objective of the present study is to evaluate the effect of acetylation on functional properties of wheat starch followed by enhance the applicability of wheat starch.

MATERIALS AND METHODS

Materials

Wheat grains were purchased in Ado Ekiti, from the popular King's Market, Ekiti State, Nigeria, in January 2016.

Extraction of Starch

The dirt in the wheat were handpicked, dried in the sun for some days. The dried seed were then grinded into flour, stored in refrigerator until further use the blended sample (flour) of the wheat (1 kg) was soaked in 10 L of water for 5 h at room temperature. The extract was centrifuged, the supernatant was decanted, 5 L of water was added to the residue and the pH was adjusted to 11 with 1M NaOH and then blended for 30 min. The sample was centrifuged, the supernatant was decanted, and the residue was blended for 15 min with 10 L of 0.03 M NaOH. The resultant was re-slurred in water and sieved with muslin cloth. The starch was allowed to settle for 30 minutes and the supernatant decanted off. Further rinsing of the starch with water, settling the starch granules and decantation of supernatant removed soluble impurities. This process was repeated until the supernatant was clear.

Acetylation of starch

The method of Salte and Salunkhe (1981) was used. 100 g of starch was dispersed in 500 ml of distilled water, the mixture was stirred magnetically for 20 min. The pH of the slurry obtained was adjusted to 8.0 using 1M NaOH. Acetic anhydride (10.2 g) was added over a period of 1h, with a stable pH range of 8.0 -

8.5. The reaction proceeded for 5 min after the addition of acetic anhydride. The pH of the slurry was adjusted to 4.5 using 0.5 M HCl. It was filtered, washed four times with distilled water and air-dried for 48 h.

Infrared spectroscopic analysis

FT-IR spectra of the native and acetylated wheat starches were obtained using shimadzu Model FT-IR – 8201 PC. The infrared spectral analysis was done to determine the functional groups of both the native and acetylated wheat starches responsible for their different functional properties. As chemical bonds absorb infra-red energy at specific frequencies (wavenumbers), the basic structures of compounds could be determined by the spectral locations of their IR absorptions.

Determination of Functional Properties

Swelling power (SP)

The method described by Daramola *et al.,* (2006) was used to determine the swelling power with slight modifications. The starch sample (0.1 g) was weighed into a test tube and 10 ml of distilled water was added. The mixture was heated in a water bath at a temperature of 50^{O}C for 30 min with continuous shaking. In the end, the test tube was centrifuged at 1500 rpm for 20 min in order to facilitate the removal of the supernatant which was carefully decanted and weight of the starch paste taken. The swelling power was calculated as follows:

$$\text{Swelling power} = \frac{\text{Weight of starch paste}}{\text{Weight of dry starch sample}}$$

This was carried out over a temperature range of 50^{O}C – 100^{O}C.

Gelatinization temperature (GT)

This was evaluated using the method of Attama *et al.,* (2003). 1 g of the starch sample was put in a 20 ml beaker and 10 ml of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was then read with a thermometer suspended in the starch slurry.

Water absorption capacity (WAC)

The method of Omojola *et al.,* (2010) was used with slight modifications. 5 % w/v of the starch sample was dispersed in a pre-weighed centrifuge tube. The tube was agitated in a vortex mixer for 2 min. The supernatant was then discarded and the weight of the tube and hydrated sample taken. The weight was calculated and expressed as the weight of water bound by 100 g dry starch.

Oil absorption capacity (OAC)

Executive oil (5ml) was added to one gram of sample in 10 ml conical flask and transferred into centrifuge tube. The mixture was stirred with glass rod to disperse the sample in oil. After holding for a period of 30 min, it was centrifuge for 30 min and the volume of separated oil noted. The oil absorbed was expressed as the percentage oil bound by 100 g samples. The density of oil (0.93 g/cm^3) was determined (Sathe and Solunkhe, 1980).

$$\% \text{ OAC} = \frac{\text{Mass of oil absorbed}}{\text{Weight of sample}} \times 100$$

Foaming capacity (FC)

The method of Omojola *et al.,* (2010) was used with slight modifications. One gram of starch sample was homogenized in 50 ml distilled water using a vortex mixer (vortex 2 Genie set at shake 8) for 5 min. The homogenate was poured into a 100 ml measuring cylinder and the volume recorded after 30 sec. The foam capacity was expressed as the percent increase in volume.

ISSN: 0975-8585

Emulsion capacity (EC)

The method of Omojola *et al.,* (2010) was also used with slight modifications. 1 g sample was dispersed in 5 ml distilled water using a vortex mixer for 30 sec, after which 5 ml of vegetable oil (groundnut oil) was added gradually and the mixing continued for another 30 sec. The suspension was centrifuged at 1600 g for 5 min. The volume of oil separated from the sample was read directly from the tube, while the Emulsion capacity is the amount of oil emulsified and held per gram of sample.

Statistical analysis

An analytical determination for the native and acetylated starches was carried out in triplicates and standard deviations were noted. The data was subjected to one way ANOVA to analyze the significant difference in all data and Duncan's Multiple Range Test (DMRT) ($P \leq 0.05$) to analyze the significant difference between mean values of samples using SPSS 18 software (SPSS Institute Inc., Cary, NC, USA).

RESULTS AND DISCUSSION

The FT-IR analysis of native and acetylated wheat starches are shown in **fig. 1** and **2**. The native wheat starch showed a typical broad hydroxyl peak around 3412.08 cm^{-1}. On acetylation, the FT-IR spectrum showed a new peak around 1242.16 $cm^{-}1$ for the acetylated.

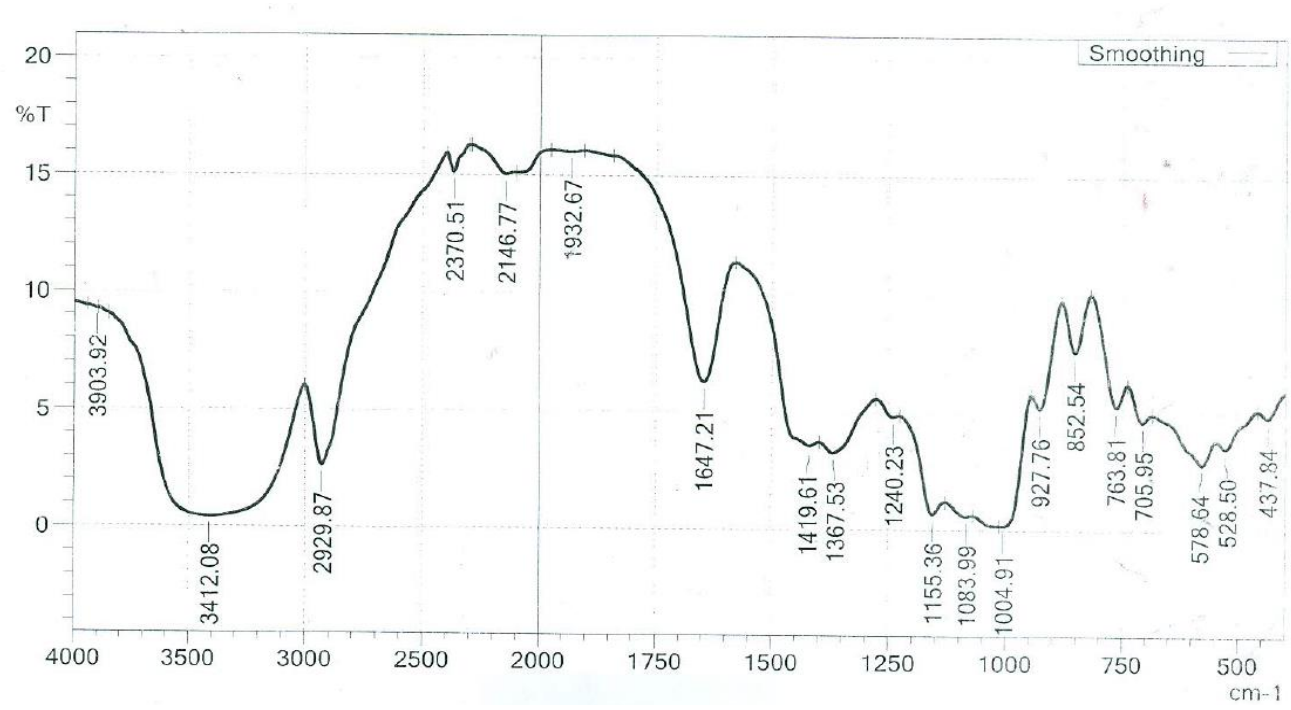
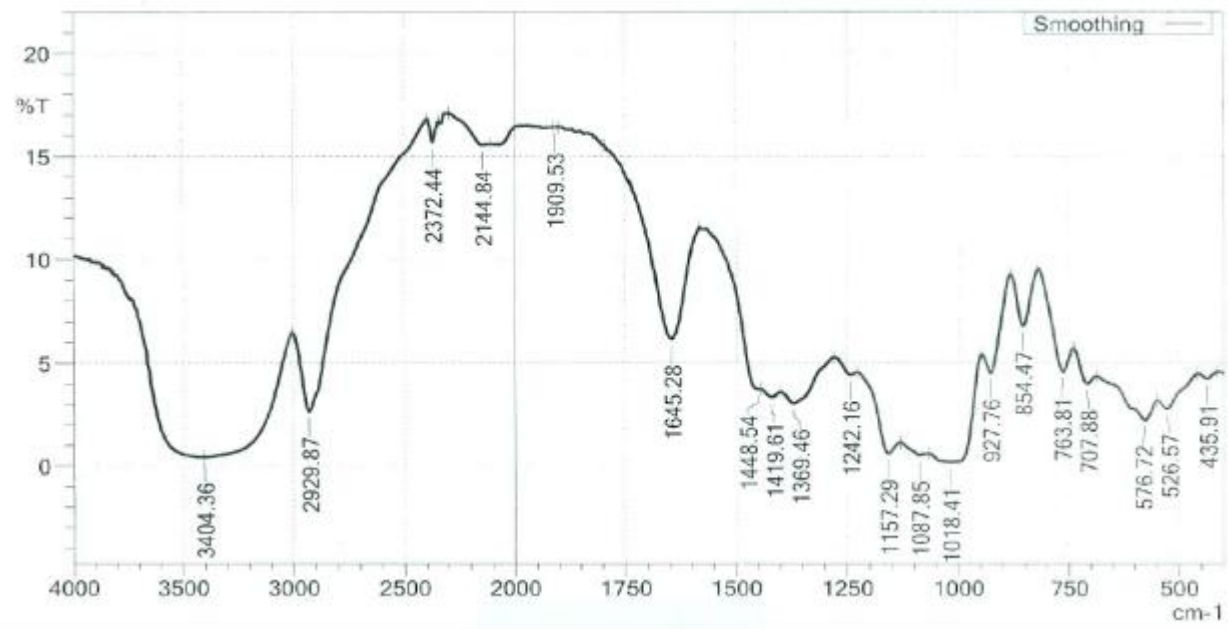


Fig 1: FTIR result of native wheat starch

ISSN: 0975-8585



The WAC of native and acetylated starches is shown in **Table 1**.

Table1: FT-IR data of native and acetylated wheat starch

Type of bond	Frequency before Acetylation (cm^{-1})	Frequencyafter Acetylation (cm^{-1})
O-H (Hydroxyl)	3412.08	3404.36
C-O (Acetate)	1240.23	1242.16

Results shows that modification process increased the water absorption capacity in acetylated (80%) starches compared to that of native (70%) starch. The modification process using chemicals improved the water absorption capacity could be due to the addition of functional groups on starch molecules which enhanced the ability of water binding capacity for starches. The acetylated wheat starch has the highest value of oil absorption (130%) compared to native wheat starch (120%). This shows that hydrophilic and hydrophobic tendency of native wheat starch were increased after acetylation (Lawal 2004).

Foam and emulsion capacities of the acetylated starch were just slightly higher than the values obtained for the native starch suggesting that acetylated starch could find application as an emulsifier in the food industries (Ihegwuagu *et al*., 2009).

The swelling profile of wheat starch compared with that of acetylated wheat starch over a temperature range of 50 – 100^{O}C is shown in **fig 3**. The swelling profile shows a general trend of increase with increase in temperature which is more uniform in the unmodified starch than in the modified starch (**Table 2**). This is an indication of the water absorption characteristic of the granules during heating. The modified starch at lower temperatures of 50 – 70^{o}C was observed to have higher swelling capacity than the unmodified starch, while the swelling power of the unmodified starch experienced an increase in swelling power at higher temperatures. Since increase in swelling power is indicative of suitablity of a starch being used as a disintegrant in the pharmaceutical industry (Chowdary *et al.*, 2011), hence, it appears that acetylated wheat starch might be a better choice as a disintegrant in the formulation of tablets especially since tablets are compounded and used at the temperature range where it has a better swelling. Also high swelling power results into high digestibility and ability to use starch in solution suggesting improved dietary properties and the use of starch in a range of dietary applications (Nuwamanya *et al*, 2010).

ISSN: 0975-8585

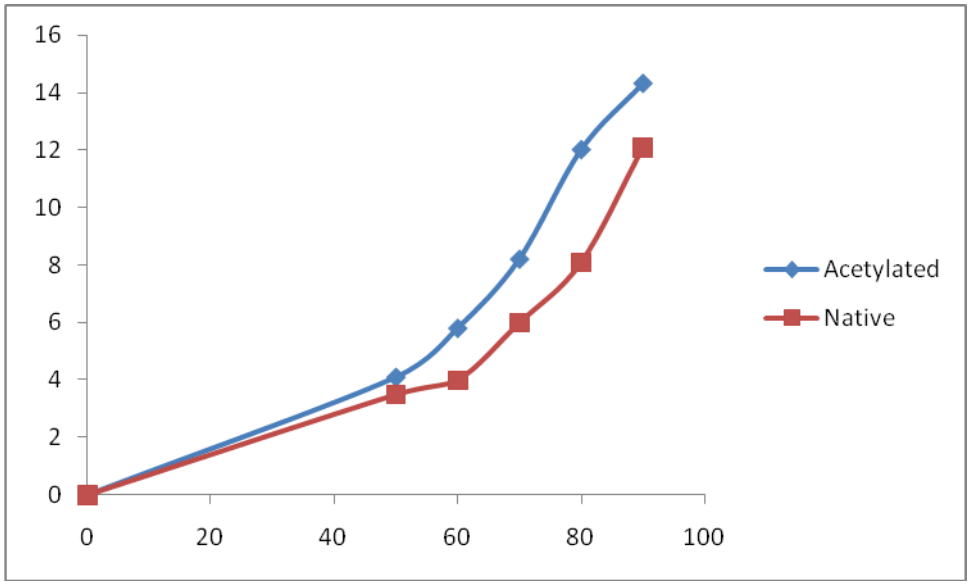


Fig 3: FTIR Result of acetylated wheat starch: Swelling properties of native and acetylated wheat starches

The native starch sample was observed to have a gelatinization temperature of 70^{O}C which falls within the range of gelatinization temperatures commonly observed for starches (**Table 2**).

Table 2: Functional properties of native wheat starch and acetylated wheat starch

Parameters	Native Wheat Starch	Acetylated Wheat Starch
Water absorption capacity (%)	70 ± 3.01	80 ± 5.02
Oil absorption capacity (%)	130 ± 2.01	140 ±3.02
Emulsion capacity (%)	24 ± 0.05	28 ± 0.02
Gelation temperature(O^{C})	70 ± 0.00	84 ±0.00
Foam capacity (%)	2.0 ± 0.00	4.0 ±0.00

This is in accordance with the value observed by Agbo *et al.*, 2010. The modified starch (acetylated) gelatinize at higher temperature (70^{o}C), this indicates that industries that require the use of starch in the gel form might not find this modified starch very useful compare to the native starch.

CONCLUSION

This work has compared the functional properties of acetylated wheat starch (modified) and the native unmodified wheat starch. This highlighted some important effects of acetylation modification on the functional properties of wheat starch and these properties shows better swelling, oil absorption, foaming capacity, emulsion capacity and water absorption properties over the native starch indicating that acetylated wheat starch is a potential source of industrial starch. The good swelling power makes it a promising pharmaceutical excipient and the good emulsifying properties suggesting that acetylated starch could find application as an emulsifier in the food industries. This type of starch from a non-conventional source will reduce the cost of producing starch and eliminate or minimize competition on stable food crops like cassava or potatoes.

ACKNOWLEDGEMENT

The authors of this manuscript hereby without missing words appreciate the contributions of members, Chemical Science Dept., ABUAD in various capacities, where larger portion of this work was carried out.

ISSN: 0975-8585

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