



Acetylated Starch Prepared from Sweet Potato (*Ipomoea batatas* L): Functional and Pasting Properties.

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Abstract

The starch from sweet potato (*Ipomoea batatas* L) was extracted and then modified through acetylation with acetic acid to produce acetylated sweet potato starch. The native and modified starches were analyzed for their functional characteristics and pasting properties, and were also characterized using Fourier Transform Infrared (FTIR) Spectroscopy and Scanning Electron Microscopy (SEM). The results indicated that after modification, the contents of moisture, ash, protein, fat, and fiber were diminished. The acetylation process led to an increase in swelling power, solubility, and the capacity for water and oil absorption in the starch, while it exhibited the lowest gel concentration, reduced syneresis, and enhanced transparency. The acetylated starch demonstrated a decrease in pasting temperature, setback, and final viscosity values. An FTIR band at 1792 cm⁻¹ confirmed the successful formation of acetylated starch via the esterification process. The SEM image indicated that the acetylated starch exhibited agglomeration of the starch granules. These findings underscore the significant potential of acetylated sweet potato starch in the production of confectioneries, salad cream, mayonnaise, texturizing agents, thickeners, stabilizers, and fillers for beverages and baked goods, while also contributing to industrial energy and time savings.

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Introduction

Starch is one of the essential elements that have been extensively used for both food and non-food applications due to its numerous properties and low cost. Recently, starch and its derivatives are widely employed in the food industry as carrier materials to protect and act as encapsulating agents for volatile substances (Robert *et al.*, 2012; Gao *et al.*, 2014; Najafi *et al.*, 2016; Wang *et al.*, 2016; Acevedo-Guevara *et al.*, 2018). Carrier materials operate as storage tool to safeguard sensitive substances such as flavors, oils, minerals, vitamins, and food colors that are sensitive to light, oxidation or high temperature (Belingheri *et al.*, 2015; Wang *et al.*, 2016; Hoyos-Leyva *et al.*, 2018; Lei *et al.*, 2018). However, the efficacy of starch as a carrier to control the release rate of components is limited due to its low surface area and pore volume on the granules (Zhu, 2017; Yang *et al.*, 2018). Therefore, modification treatments are routinely applied to the surface of starch granules to fulfill their intended functionalities. Chemical modification generally results in altered intra- and intermolecular bonding, resulting in changed physicochemical and functional properties of starches (Iftikhar and Dutta 2020). Chemical techniques encompassing oxidation, esterification and etherification involve bonding association of reactive

sites of hydroxyl groups of starch with new chemical groups, altering the chemical nature of the starch matrix (Wang *et al.*, 2021).

Acetylation is one of the chemical treatments that have been utilized to change starch molecules to better their application for encapsulating purposes (Robert *et al.*, 2012; García-Tejeda *et al.*, 2015; Najafi *et al.*, 2016; Acevedo-Guevara *et al.*, 2018). Treating native starch granules with acetic anhydride (AA) or acetic acid assists in expanding the binding sites on the starch granules' surface (García-Tejeda *et al.*, 2015) and altering the hydrophilic character of the starch molecules into somewhat hydrophobic. This treatment comprises the substitution mechanism where the free hydroxyl (OH) groups in the starch chains are replaced by acetyl (CH₃COO) groups. The CH₃COO groups placed into the starch granules allow volatile chemicals to interact with the starch molecules. Because of this, the acetylated starch is viewed as a potential alternative for the development of surface-modified starch, which helps to improve the binding sites on the starch granules for wider applications as compared to the native starch.

Therefore, the main purpose of this study was to investigate the effects of acetylation treatment on the surface-modified sweet potato starch. This study will provide useful information for future research to

increase the usage of surface-modified starch in the food industry.

Materials and Methods

Materials

In this study, the following materials were used: sweet potato tubers, muslin cloth, sieve, distilled water and food processor and olive oil.

Starch Extraction

The starch was extracted from sweet potato tubers using the method described by Senanayake *et al.* (2014). The sweet potato tubers were rinsed under running water to eliminate attached soil and subsequently peeled by hand. Subsequent to peeling, the sweet potatoes were diced into small fragments and processed in a food processor with the addition of clean water for 3-4 minutes at medium speed. Subsequent to sieving, water was introduced to the residue for further extraction. The filtrate was passed through muslin fabric for filtration. The starch slurry will be permitted to settle for 2 to 3 hours at ambient temperature. The supernatant was decanted. The starch at the bottom of the container was resuspended in water, filtered, and allowed to remain undisturbed for two hours. The settlement procedure was conducted thrice. The sediment starch was desiccated in an oven at 50°C for 6-8 hours and thereafter allowed to cool to ambient temperature. Dried starch was milled and put through a sieve. The recovered starch was stored in an air-tight container and labeled NSPS for later analysis. The starch yield was calculated as:

$$\text{Starch Yield} = \frac{\text{Extracted Starch}}{\text{Amount of sweet potatoes}} \times 100$$

Starch Modification

Acetylation of Starch: The chemical modification of starch using acetic acid was done according to the method of Granza *et al.*, (2015). The alteration was done by dispersing 250g of the starch sample in 1000mL distilled water and the pH was changed to 10 using 1M NaOH. The mixture was stirred and continued for 20min at room temperature, 45mL of 4% acetic acid was added drop-wise with simultaneous adjustment of pH. After permitting the reaction for 5min, 1N HCl was used to decrease the pH to 4.5, which terminated the reaction. The residue was collected, rinsed five times with distilled water and dried. The dried sample was processed, stored and labeled ASPS for later investigation.

Acetyl Group Percentage and Degree of Substitution

1g acetylated starch was added to 50 mL 75% ethanol in a stoppered flask and heated at 50°C for 30minutes and cooled to room temperature. 40ml of 0.5M KOH

was added and occasionally swirled for 72h. The mixture was then titrated against 0.05M HCl using phenolphthalein indicator. The titrated solution was allowed to stand for 2h, enabling the release of more alkali, and titrated again. Native starch was utilized as a reference. (Colussi *et al.*, 2015).

$$\text{Degree of Substitution} = \frac{\text{ACG} (\%) \times \text{Sample Weight}}{4300 - (42 \times \text{ACG})} = \frac{162 \times \text{ACG}}{4300 - (42 \times \text{ACG})}$$

Characterization of Native and Acetylated Starches

Infrared Spectroscopy: The infrared spectra of native and modified starch samples were obtained using a Fourier Transform Infrared (FTIR) spectrometer (Cary 630, Agilent Technologies, USA) operating at a resolution of 2.0 cm⁻¹ with KBr optics within a wave number range of 650 – 4000 cm⁻¹

Scanning Electron Microscopy: The native and modified starch samples' morphology was observed using a scanning electron microscope (SEM, EVO-18, Zeiss, Germany). Samples were vacuum dried, fixed on double-sided tape, sputter-coated with gold and observed at 5000x magnification.

Swelling Power and Solubility: The swelling power and solubility were examined according to the method published by Paramitasari *et al.* (2024) and Dhull *et al.* (2021). 0.6 g of the starch samples was cooked with 40 mL of water at 50, 60, 70, 80 and 90°C for 30 mins. Lump formation was prevented by stirring. The suspension was centrifuged at 3000 rpm for 15 min. Supernatant was thoroughly removed and the starch sediment was weighed. An aliquot of supernatant (5 mL) was collected in a pre-weighed petri dish and evaporated for 2 h at 110°C and then weighed. The residue collected after drying of the supernatant represents the amount of starch solubilized in water. The result is expressed as:

$$\text{Solubility} = \frac{\text{weight of soluble starch}}{\text{weight of sample}} = \frac{\text{Swelling Power}}{\frac{\text{weight of sediment paste}}{\text{weight of sample} - \text{weight of soluble starch}}}$$

Water and Oil Absorption Capacities

The water absorption capacity of the starch samples was evaluated according to the method of Claver *et al.* (2010). 10 mL of distilled water was mixed with 1 g of starch samples and the mixture was centrifugation at 4000 rpm for 10 min. The water absorption capacity was stated as:

$$\text{Water Absorption Capacity} = \frac{\text{weight of residue (g)}}{\text{weight of sample (g)}}$$

The oil absorption capacity of the starch samples was measured according to the method of Ogundiran and Ogundiran (2024). 10 mL of olive oil was combined with 1 g of starch sample and was allowed to remain at room temperature for 30 min, then centrifuged at 3000 rpm for 15 min. The oil absorption capacity was calculated using the equation as follows:

$$\text{Oil Absorption Capacity} = \frac{\text{weight of residue (g)}}{\text{weight of sample (g)}}$$

Paste Clarity: The paste clarity of the samples was investigated using the approach of Bhandari and Singhal (2003). 50 mg of the starch samples were suspended in 5 mL of distilled water in test tubes. The test tubes were then cooked in a boiling water bath (with intermittent shaking) for 30 min. After cooling to room temperature, the % transmittance was evaluated at 650nm against a water blank using a spectrophotometer. The starch samples were kept at 30°C for 1-5 days measuring the transmittance at 650nm at 24h interval.

Gelation Properties: Test-tubes containing starch suspensions of 2%, 4%, 6%, 8%, 10% and 14% (w/v) of starch in 5 mL distilled water were heated for 1 h in boiling water followed by rapid cooling under running water. The test tubes were further cooled at 4°C for 2 h. Least gel concentration (LCG) is the concentration at which the sample did not fall or slip when the test tubes were inverted (Lin *et al.*, 2017).

Functional Properties

Swelling Power and Solubility

The swelling power values of native and acetylated sweet potato starches ranged from 1.52 to 3.12 and 2.01 to 4.08, respectively, with a stepwise rise in temperature from 50°C to 90°C. The swelling power is an indication of the ability of the starch to hydrate under specified cooking circumstances. The swelling power of starch depends on the capacity of starch molecules to hold water by hydrogen bonding and is regulated by a robust micellar network, amylopectin molecular structure and amylose concentration (Li and Vasanthan 2003; Tang *et al.*, 2005). Table 1 demonstrated an increasing swelling power of all the starch samples as the temperature increased. This is because the binding forces of the starches were decreased as a result of an increase in temperature, thus facilitating the entry of water into the crystalline area of the starches with a subsequent increase in the

Pasting Properties: The pasting properties of the starch samples were measured using a Rapid Visco Analyzer (Newport Scientific Pty Ltd, Warriewood NSW 2102, Australia). Starch samples (3 g) were weighed into a previously dried canister and 25 mL of distilled water was dispensed into the canister containing the starch samples. The suspension was thoroughly mixed and the canister was fitted into the Rapid Visco Analyzer as recommended. Each suspension was kept at 50°C for 1min and then heated up to 95°C with a holding time of 2 min, followed by cooling to 50°C with 2 min holding time. The rate of heating and cooling was at a constant rate 11.8°C/min. Pasting parameters namely, pasting temperature, peak viscosity, trough, breakdown, final viscosity and setback viscosity, were recorded. All results of the analysis for this study were determined in triplicates.

Results and Discussion

Degree of modification and Percentage of Substituent groups. The Acetyl group percentage (ACG) of Acetylated Sweet Potato Starch (ASPS) with the value of 0.82% was below the maximum recommended value of 2.5% (Food and Drug Administration). Lee *et al.*, (2018) similarly argue that acetyl substitution favorably occurs in the amorphous portion of amylopectin rather than the usually amorphous amylose. The degree of substitution of Acetylated Sweet Potato Starch having the value of 0.03.

swelling power of the starches. Acetylated starch has a higher swelling power value than native starch and this can be related to an increase in long chains of amylopectin and is in agreement with the results of Shaari *et al.* (2021)

The solubility index values of the starches ranged from 0.06 to 0.36 and 0.11 to 0.45 for native and acetylated sweet potato starches, respectively. Solubility corresponds to hydrophilicity and amylose content. Table 1 showed that there was an increase in the solubility of all the starches as the temperature increased and these results are in line with previous studies that swelling power and solubility of starch will increase after a chemical modification process, as reported by Das *et al.*, (2010) for white yam starch.

Table 1: Swelling power and Solubility indexes of native and acetylated sweet potato starch at varied temperature.

Samples/Temp	50°C	60°C	70°C	80°C	90°C
Swelling Power					
NSPS(g/g)	1.52±0.05	1.82±0.02	2.10±0.02	2.74±0.01	3.12±0.02
ASPS(g/g)	2.01±0.03	2.42±0.02	3.19±0.02	3.82±0.03	4.08±0.03
Solubility Index					
NSPS(g/g)	0.06±0.01	0.10±0.01	0.19±0.01	0.30±0.01	0.36±0.03
ASPS(g/g)	0.11±0.0	0.15±0.02	0.24±0.01	0.37±0.01	0.43±0.02

Values are expressed as mean ± standard deviation of triplicate determination; NSPS = Native sweet potato starch, ASPS = Acetylated sweet potato starch

Water and Oil Absorption Capacities

The effects of acetylation on the water and oil absorption capacity of sweet potato starches are demonstrated in Table 2. The results demonstrate that the water and oil absorption capacity of the starch improved following the changes with acetylated starch having the highest value of 1.74g/g against 1.52g/g observed for native starch. According to Ikegwu *et al.*, (2010), water absorption capacity is a result of various aspects, including size, shape, conformational properties, steric factors, lipids and carbohydrates

associated with proteins and others. Ikegwu *et al.*, (2009) also showed that the ability of food to absorb water and oil may help to enhance sensory qualities such as flavor retention and mouth-feel. The improvement in the water and oil absorption capacity of the modified starches is a result of the introduction of functional groups on the starch molecules which facilitate a more enhanced binding capacity than the native starch and these results was in accordance with the previous work of Lawal (2004).

Table 2: Water and Oil Absorption capacity of the native and modified sweet potato starches.

Samples	WAC(g/g)	OAC(g/g)
NSPS	1.52±0.01	1.73±0.02
ASPS	1.74±0.02	2.03±0.05

Values are expressed as mean ± SD of triplicate determinations WAC = Water Absorption Capacity, OAC = Oil Absorption Capacity, NSPS = Native sweet potato starch, ASPS = Acetylated sweet potato starch

Paste Clarity: Paste clarity is a much-sought characteristic of starches for its utilization in food industries since it directly influences brightness and opacity in food that contains it as a thickener (Mweta, 2009). The influence of storage days on the paste clarity of the sweet potato starches was shown to decrease in all samples. A similar time-dependent drop in % transmittance has been found for banana starch by Bello-Perez *et al.*, (2000). Table 4 illustrates the % transmittance between the modified starch sample and native starch sample. The results showed that the

starch paste clarity was enhanced after modification. The high transmittance value of acetylated starch can be attributed to the chemical substitution of the hydroxyl group in the starch molecule, which causes repulsion between adjacent starch molecules and reduces inter-chain association which facilitates improved percentage transmittance (Lawal, 2004). The decline of % transmittance of the NSPS is a retrogradation trend and this agrees with findings by Lawal (2004)

Table 3: Effect of storage time on transmittance of native and modified sweet potato starches Samples

/ Days	1	2	3	4	5
NSPS (%)	95.3±0.20	93.7±0.15	91.7±0.20	87.3±0.21	84.5±0.20
ASPS (%)	96.9±0.20	95.7±0.98	94.3±0.40	90.2±0.21	88.5±0.10

Values are expressed as mean ± standard deviation of triplicate determination; NSPS = Native sweet potato starch, ASPS = Acetylated sweet potato starch

Gelation Properties: The ability of starch to form a gel is a desirable quality in food industries. The least gel concentration (LGC) of native and acetylated sweet potato starches are 10 and 8 (w/v), respectively. The gelation properties are influenced by a physical competition for water between protein gelation and starch gelatinization (Singh and Singh, 2001). The modified starches had a lower least gel concentration

when compared to native starch. Similar results were reported for arrowroot starch by Okoye *et al.*, 2010. This showed that modified starches are purer than the native starch; hence, competition for water between protein and starch is greatly reduced in modified starches. Table 6 shows the lowest gel concentration of the native and modified starch samples

Table 4: Least gel concentration of the native and modified sweet potato starches

		Sample concentration (%w/v)						
Samples	Remark	2	4	6	8	10	12	14
NSPS	Gelation	-	-	+	+	+	+	+
	State	Liquid	Liquid	Viscous	Viscous	L.G.C	F.G	F.G
ASPS	Gelation	-	-	+	+	+	+	+
	State	Liquid	Liquid	Viscous	L.G.C	F.G	F.G V.F.G	

L.G.C = Least Gel Concentration, F.G = Firm Gel, V.F.G = Very Firm Gel, NSPS = Native sweet potato starch, ASPS = Acetylated sweet potato starch

Pasting Properties: Table 5 displays the pasting qualities of native and acetylated sweet potato starches. The application of heat in the presence of water changes the behavior of starch and starch-based products, resulting in various pasting profiles. Pasting qualities of starch are significant functional properties linked to the ability of starch to operate in a paste-like way (Ojo *et al.*, 2022; Tsakama *et al.*, 2010). The pasting properties are used to anticipate the behaviour of starch during and after cooking. There is always and clear association between peak viscosity, swelling power and breakdown viscosity; additionally higher amount of amylopectin is connected with high swelling capacity (Ashogbon and Akintayo 2014). It can be deduced from the table that the peak viscosity of the modified starch was higher as compared to native starch, as was also demonstrated by improved swelling as reported by Lie *et al.*, (2018), with acetylated starch having the greatest value of peak viscosity. Peak viscosity is commonly connected with the end product and also offers an indicator of viscous loads anticipated to be encountered during mixing. After acetylation, there was a reduction in the pasting temperature, setback, and final viscosity parameters of the starch samples. This reduction following acetylation was also reported by Kinn-Kabari (2015) for arrowroot starch. The pasting temperature which is the minimal temperature required to cook a given starch of food sample. This temperature is higher than the gelatinization temperature, implying that starches are gelatinized before rise in viscosity (Kiin-Kabari,

2015), Rosida *et al.*, 2017). Low pasting temperature of the acetylated starch (78.8°C) indicated that the starch would be easier to cook and would require less heat for gelatinization to start. The final viscosity, which is the ability of starch material to form a viscous paste or gel after cooling, was lowered in acetylated starch. The data indicates that there was a conformational reordering and rearrangement prompted by the acetylation modification process limited the affinity of the hydroxyl group of one molecule for another with the introduction of other functional groups. The introduction of functional groups to replace the hydroxyl groups restricts production of such binding forces and consequently accounts for the reduction in final viscosity of acetylated starch relative to native starch and this is in keeping with results by Lawal (2004) for white yam starch. The breakdown value for acetylated starch (3963±18.3) was substantially higher than native starch (3788±21.9), suggesting greater disintegration and decreased tendency for setback. The differences in the breakdown value of the starches can be ascribed to the granule stiffness, the temperature and the degree of mixing and shear applied to the samples (Singh *et al.*, 2003). Low value of breakdown viscosity reflects greater stability of starches under shear stress and hot temperatures (Ezeocha and Okafor, 2016). Hence, a higher breakdown viscosity leads in reduced ability of the starch to survive heating and shear stress during cooking (Adebowale *et al.*, 2005). The pasting time which is the measure of the cooking time for the

starch, was shorter in acetylated starch (4.27 ± 0.09), compared to the native starch (4.44 ± 0.09). This indicates that acetylated starch would have little resistance to swelling and as such, would be predicted to swell rapidly and become prone to concurrent shear-

driven breakdown than starches. Therefore, the acetylated starch sample with the shortest pasting time and lowest pasting temperature will yield larger gains in time and energy than the native starch sample when utilized in food preparations.

Table 5: Pasting properties of native and acetylated sweet potato starches.

Samples	PV	T	RVA Parameters (cP)				$P_{temp} (^{\circ}C)$	$P_{time} (min)$
			BD	FV	SB			
NSPS	6947 ± 7.70	3159 ± 15.5	3788 ± 21.9	3948 ± 21.9	825.5 ± 6.36		80.07 ± 1.27	4.44 ± 0.09
ASPS	7080 ± 8.49	3117 ± 9.89	3963 ± 18.3	3939 ± 19.5	817.5 ± 12.4		78.8 ± 0.64	4.27 ± 0.09

Values are expressed as mean \pm SD.

PV = Peak Viscosity, T= Trough, BD= Breakdown, FV= Final Viscosity, SB= Setback Viscosity, P_{temp} = Pasting Temperature, P_{time} = Pasting Time SPS = Native sweet potato starch, ASPS = Acetylated sweet potato starch

Infrared Spectroscopy: Fig.1 shows the FTIR spectra for Native and Acetylated sweet potato starches. Native starch spectrum showed a broadband at around 3300cm^{-1} , indicating the presence of the O-H group stretching which decreased after modification. The bands at 1353cm^{-1} and 2970cm^{-1} are attributed to O-H bending and C-H stretching, while the band at $1250\text{--}970\text{cm}^{-1}$ has been assigned to C-O stretching. Other absorption peaks that appear at 995cm^{-1} , 1077cm^{-1} ,

1148cm^{-1} are associated with the stretching of the C-O bond. The FTIR spectrum of the acetylated starch shows a absorption peak of C=O band vibration at around 1742cm^{-1} indicating that acetylated starch has been successfully formed through esterification reaction. The band at 1341cm^{-1} has been assigned to C-H stretching of acetyl groups. The band at 1587cm^{-1} has been assigned to the bending vibration of CH_2 and the stretching of C-O of the acetyl group.

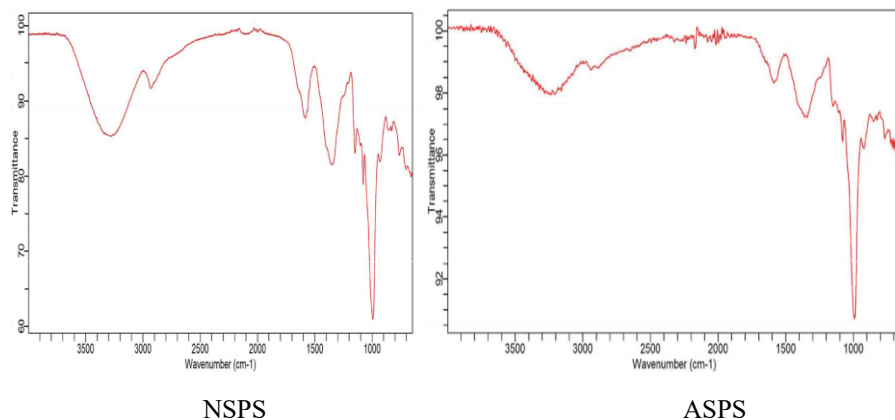
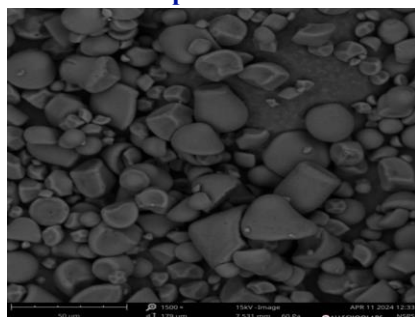


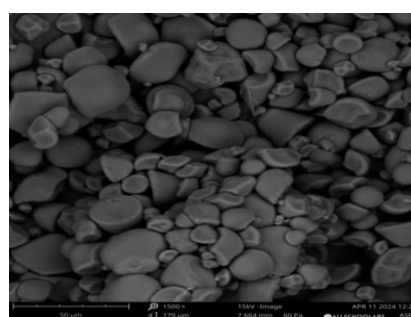
Figure 1: FTIR Spectra for Native and Acetylated Sweet Potato Starches

Scanning Electron Microscopy: The SEM micrograph showed that native sweet potato starches exhibit oval to rod-shaped granules, which is smooth with very minimal damage; this suggests that the method of extraction and drying did not cause significant damage to the starch and this result corroborates with previous works of Kim *et al.* (2020). Tupa *et al.* (2013) reported that the surface of acetylated starch granules was rougher than that of native starch. The alteration of starch by acetylation can induce the deformation of starch granules, which

causes the formation of small fragments. The granules of acetylated starch displayed a varied degree of surface damage, porosity and fragmentation. The incorporation of acetyl groups into starch molecules diminishes the inter- and intramolecular bonds of starch, disintegrating starch granules (Zhang *et al.*, 2009). The integrity of the starch diminishes, and its structure becomes progressively porous during the acetylation reaction; subsequently, the starch granules undergo fragmentation and aggregate with one another (Collusi *et al.*, 2015)



NSPS



ASPS

Figure 2: SEM images of Native and Acetylated Sweet Potato Starches

Conclusion

The research concentrated on the analysis of the proximate composition, functional properties, pasting properties as well as characterization of the native and acetylated sweet potato starches. The starch samples exhibited differences in the proximate composition, functional properties and pasting properties that differentiated them into specified roles for applicable utilization in the manufacture of confectioneries, thickeners stabilizers, binders, fillers, flavoring agents, texturizers, cheese, gravies, sauces, coating system, weaning foods, gelling agents, composite flours, energy-giving foods, bakery foods, dairy products, drugs, beverages and brewery products.

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