Effects of reactive extrusion
on cross-linking of corn starch

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Abstract

The present work aimed to evaluate the efficiency of the dual modification of corn starch by reactive extrusion, using a mono-thread extruder, and also sodium tripolyphosphate and trimetaphosphate to obtain cross-linked starch. Native starch, extruded native starch, and starch cross-linked by conventional methods were used as standards to evaluate the modification efficacy by reactive extrusion. The content of phosphorus residues allowed by legislation in starch modification is 0.4%. Therefore, the reactive extrusion demonstrated greater effectiveness because cross-linked starch obtained by this method was within the established limit of phosphorus residues, in contrast to that obtained by the conventional method, which exceeded this amount. In addition, it required greater water consumption for residual removal, therefore producing effluents, and also wasting energy and time. Cross-linking by the extrusion process showed a profound influence on the characteristics of modified starch, generating specific functional characteristics, mainly in terms of pasting properties. Also, the samples presented low viscosity and pasting temperature. In conclusion, cross-linking through reactive extrusion was fast, produced starch with phosphorus content within the limits allowed by legislation, and generated different physicochemical characteristics.

Introduction

Starch is a natural polymer present in plants in the granular form, and found in abundance in cereals, legumes, tubers, and roots. Starch, especially corn starch, is used extensively in the food industry due to its high global production and widespread availability, and the main source of carbohydrates and energy for human.

The use of native starch by the food industry is limited; its natural characteristics do not support many thermal processes and pH changes. In addition, native starch is inert, insoluble at room temperature, and highly resistant to enzymatic hydrolysis, thus generating technological problems for processing, and therefore requiring specific functional properties (Ashogbon and Akintayo, 2014). To adapt to the limitations of native starches and introduce specific

functionalities, certain chemical, physical, or enzymatic modifications are made, alone or in combination. Due to these modifications, it is possible to improve properties such as texture, transparency, and the gloss of the starch gel; to reduce syneresis and retrogradation; and to increase the freeze-thaw stability; modify its cooking properties (Ashogbon, 2021).

Substitution and cross-linking are the two main types of modification. Phosphorylation is a reaction that can be used in the substitution and cross-linking modifications of starch destined for the food industry (Dong and Vasanthan, 2020). Substitution is conducted through esterification at the hydroxyl of the phosphate group from the starch molecule in the amorphous region. Cross-linking occurs *via* the formation of intermolecular bonds between a phosphate and hydroxyl groups from two starch

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molecules in the amorphous region (Landerito and Wang, 2005b; Dong and Vasanthan, 2020). Phosphorylation produces monosubstituted phosphates *via* substitution reaction and/or disubstituted starch phosphates *via* cross-linking reaction, and the type of phosphorylation that occurs depends on the reaction conditions such as pH, reagent type, and temperature, among other factors (O'Brien and Wang, 2009).

Cross-linked starch has been used mainly to enhance starch viscosity and stability. In cross-linked starches, a decrease is observed in the swelling power, solubility, and transparency of the paste; it also shows greater resistance to cohesion and mechanical shear at low pH values and high temperatures (Chen *et al.*, 2017).

However, individual modification of starches is ineffective in some cases of food and industrial applications. As an example, modification through a succinylation reaction can provide desirable qualities such as high solubility in cold water, high viscosity, greater paste clarity, delayed retrogradation, and freeze-thaw stability (Moin *et al.*, 2016; Ashogbon, 2021). On the other hand, succinylated starches are unstable under high-temperature shear (Ačkar *et al.*, 2015; Ashogbon, 2021), thus requiring further modification to address these deficiencies. Aiming to further improve the properties and uses of starches, double modification could be an alternative to optimise the functionality of individually modified starches. Conventional modification technologies require many reagents for gelatinisation and neutralisation that can cause environmental contamination by effluents resulting from the reaction processes. To reduce pollution and increase production, physical modification methods are promising options (Singh *et al.*, 2010). As an alternative to conventional modification, extrusion has the advantage of high versatility, being organic, solvent-free, and not generating pollution by effluents. The extruder can be considered a HTST (high-temperature short-time) bioreactor; during the extrusion process, the starch is exposed to high pressure, heat, and shear force, which can cause gelatinisation which melts and breaks down the starch. It can convert the starch from a granular or semi-crystalline state to a highly viscous, plastic starch (O'Brien and Wang, 2009). Many studies report that reactive extrusion is commercially more viable for producing chemically modified starches than the conventional method by heating in an oven.

The temperature, pressure, and high shear force used in extrusion contribute to improved phosphorylation, thus making it a promising alternative for dual modification (Landerito and Wang, 2005b; O'Brien and Wang, 2009).

Extruders can be twin- and single-screws. The first is more sophisticated and expensive, producing excellent raw materials and food products. The second, a single-screw extruder, is simpler, less expensive, and generates good production. In this way, it is more viable for small and medium-sized companies or industries, but it has some limitations, such as a lack of temperature control. In the present work, a single-screw extruder was used to evaluate its ability to carry out reactive extrusion for the modification of corn starch, and the objective of the present work was to carry out the cross-linking of corn starch through the reactive extrusion process using a single-screw extruder to evaluate the efficiency of the combination of chemical and physical modifications in the properties of corn starch.

Materials and methods

Materials

Corn starch was obtained from a local market in Maringá, Paraná, Brazil. Sodium tripolyphosphate (STPP) FCC purchased from Labsynth (Brazil), and analytical grade sodium trimetaphosphate (STMP) purchased from Sigma-Aldrich (Brazil), were used. Extrusions were conducted in an IMBRA RX50 extruder (INBRAMAQ, Ribeirão Preto - SP, Brazil), equipped with a single screw measuring 5 cm in diameter and 20 cm in length, a screw speed of 50 rpm, and a feed rate of 12 g/s. Conventional modifications were performed in an air-circulated oven.

Sample preparation

Four samples were produced, and two of the samples did not undergo the cross-linking process. The samples produced and their respective codes were native starch (NS), conventionally cross-linked starch (CLS), extruded native starch (ENS) (without cross-linking), and extruded cross-linked starch (ECLS).

Modifications by the reticulation method were based on the methodology proposed by United States Patent (1959), with modifications by Lim and Seib (1993). The choice to use pH 9 was made according

to Muhammad *et al*. (2000). Reagents were mixed with starch according to Landerito and Wang (2005a).

CLS and ECLS samples, before oven or extrusion cross-linking, were prepared similarly. Anhydrous sodium sulphate (18.75 g) was solubilised in 375.0 mL of distilled water, and 18.75 g of STPP and 7.5 g of STMP were added. The pH of the solution was adjusted to pH 9 (CLS and ECLS) with 10% HCl or NaOH solution (m/v). Then, 375 g of corn starch were added to the solution, and the pH was adjusted again to pH 9 with 5% solution of HCl or NaOH (m/v). The total weight was adjusted to 833 g by adding distilled water so that the starch solids made up 45% of the suspension (m/v).

The suspensions were stirred for 1 h at room temperature, and dried to 10 - 15% humidity at 40°C in an air-circulated oven. From this stage on, the samples went on to modifications by oven or by extrusion.

To prepare the NS and ENS samples, native corn starch was suspended in distilled water in a 1:1 ratio (m/m). The suspension was stirred for 1 h at room temperature, and dried to 10 - 15% humidity at 40°C in a circulating air oven. From this stage on, the samples went on to modifications by oven or by extrusion. To carry out the oven cross-linking (CLS) and produce the NS control sample, the dried starch prepared earlier was heated for 2 h at 130°C in a conventional oven.

For modification by extrusion of the ENS and ECLS samples, it was necessary to perform a pre-treatment of the samples to increase the starch granulometry, according to Mendes *et al*. (2017). After pre-treatment, starches were extruded at 20% moisture by tests performed previously.

Determination of phosphorus content

The phosphorus content, P (%),present in the samples was determined following the technique described by AOAC (1995a; 1995b), using a UV/visible spectrophotometer (SP-2000UV) at 420 nm. The results were calculated from the standard curve of phosphorus as shown in Figure 1a. The substitution degree was calculated using Eq. 1 (Wongsagonsup *et al*., 2005):

$$Substitution\ degree = \frac{324P}{3100 - 96P} \quad \text{(Eq. 1)}$$

where, P = % phosphorus from starch.

Amylose content

The amylose content present in the samples was determined through the colorimetric method proposed by Martinez and Cuevas (1989), using a UV/visible spectrophotometer (SP-2000UV) at 610 nm. The results were calculated from the standard curve of amylose as shown in Figure 1b.
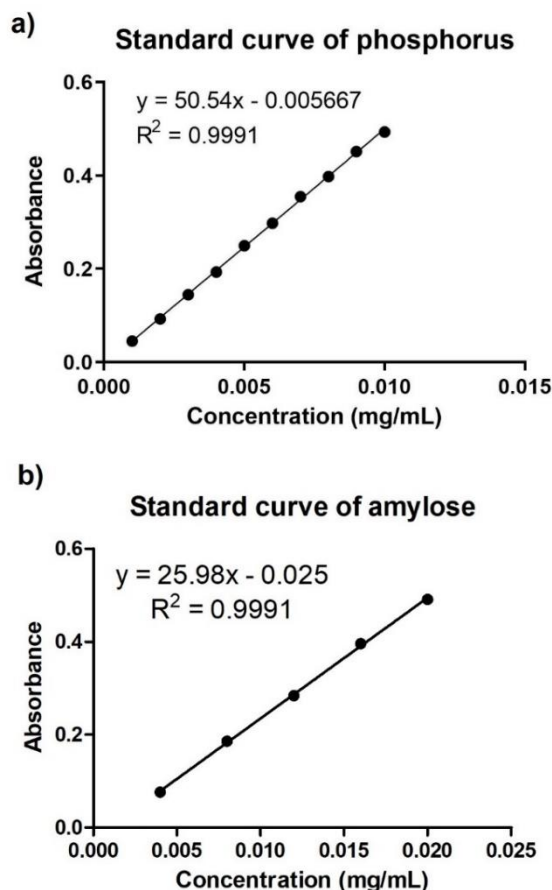

Figure 1. Standard curves of **(a)** phosphorus, and **(b)** amylose.

Swelling power and solubility

The swelling power and solubility of starches were determined according to the method described by Leach *et al*. (1959). The swelling power and solubility were calculated using Eqs. 2, 3, and 4:

$$Solubility\ (g) = initial\ supernatant\ weight\ (g) - dry\ supernatant\ weight\ (g) \quad \text{(Eq. 2)}$$

$$Solubility\ (\%) = \frac{solubility\ (g)}{sample\ weight\ (g)} \times 100 \quad \text{(Eq. 3)}$$

$$Swelling\ power\ (g/g) = \frac{sample\ weight\ (g) - sediment\ of\ sample\ after\ centrifuge(g)}{sample\ weight\ (g) - solubility\ (g)} \quad \text{(Eq. 4)}$$

Paste clarity

The paste clarity was assessed according to the method described by Morikawa and Nishinari (2000), which determines the percent transmittance (T%) at 650 nm of an aqueous starch solution (1%), subjected to heating at 95°C and stirring for 30 min, with subsequent cooling to 25°C.

Degree of gelatinisation

The degree of gelatinisation was determined following the methodology proposed by Baks *et al.* (2007). Samples (0.04 g) were dissolved in 50 mL of 0.15 M KOH, followed by centrifugation at 5,000 rpm for 10 min. One millilitre of the supernatant was removed and neutralised with 9 mL of 0.17 M HCl. Subsequently, 0.1 mL of iodine reagent (1 g of iodine and 4 g of potassium iodide in 100 mL of water) was added to form a blue complex with the dissolved amylose present in the sample. The absorbance was measured at 25°C at 600 nm (A1). The same amount of starch sample was dissolved in 50 mL of 0.4 M KOH, and boiled in a 95°C water bath for 10 min. The supernatant was neutralised with 0.45 M HCl. Following the addition of 0.1 mL of iodine reagent, the absorbance was read at 25°C and 600 nm (A2). The degree of gelatinisation was calculated by the percentage ratio of A1 (0.15 M KOH) to A2 (0.40 M KOH) using Eq. 5:

$$Degree\ of\ gelatinisation\ (\%) = \frac{A1}{A2} \times 100 \quad (Eq.\ 5)$$

Pasting properties

The pasting properties of the samples were determined according to Pumacahua-Ramos *et al.* (2015) using an RVA-4 rapid viscosity analyser (Newport Sci., Australia). Suspensions of 8% starch (m/m) in distilled water were used, and submitted to a controlled cycle of heating and cooling under constant shear (160 rpm), where they were maintained at 50°C for 2 min, heated from 50 to 95°C at 6°C/min, held at 95°C for 5 min, cooled to 50°C at 6°C/min, and held at 50°C for 2 min. Through the analysis, data on the parameters of pasting temperature, peak viscosity, trough viscosity, breakdown, final viscosity, and setback viscosity were obtained. The pasting temperature was determined by observing the initial gelatinisation temperature at the start of the gelatinisation curve. The viscosity peak was determined by observing the maximum value reached by the viscoamylographic curve. The trough viscosity was determined by evaluating the minimum viscosity after a constant temperature of 95°C was reached. The breakdown was measured as the difference between the peak viscosity and the minimum viscosity at 95°C. The final viscosity was measured by observing the maximum viscosity at the final cooling temperature of 50°C. The setback was calculated as the difference between the minimum viscosity at 95°C and the final viscosity.

Statistical analysis

The results obtained were subjected to analysis of variance (ANOVA) followed by Tukey's test at a significance level of 5% using Sisvar 5.6 software (Ferreira, 2014).

Results and discussion

Phosphorus content

Table 1 shows that the extrusion process caused a non-significant loss in phosphorus contents of NS and ENS samples. For CLS and ECLS samples, significant loss in phosphorus content was observed, and the ECLS sample had lower phosphorus content than the CLS sample. The conventional modification to which the CLS sample was subjected took a longer time, so the cross-linking reaction and the insertion of phosphate groups in the starch structure were improved. As for the combination of modifications to

Table 1. Phosphorus contents, substitution degrees, swelling power, solubility, and amylose contents of starches.

Sample	Phosphorus content (%)	Substitution degree	Swelling power (g/g)	Solubility (%)	Amylose content (%)
NS	0.20 ± 0.01^c	0.0215	8.38 ± 0.18^b	1.02 ± 0.14^d	40.31 ± 5.50^{ab}
CLS	0.52 ± 0.03^a	0.0551	9.74 ± 0.31^a	5.24 ± 0.17^c	33.69 ± 0.38^b
ENS	0.16 ± 0.01^c	0.0171	5.11 ± 0.02^c	21.45 ± 1.85^b	47.51 ± 0.22^a
ECLS	0.42 ± 0.01^b	0.0442	10.33 ± 0.16^a	36.99 ± 1.18^a	37.34 ± 1.96^{ab}

Similar lowercase superscripts indicate no statistical difference between the samples based on Tukey's test ($p > 0.05$).

which the ECLS sample was subjected, the reaction occurred quickly enough to promote the cross-linking and insertion of phosphate groups in the starch structure; however, the level of modification in this sample was lower as compared to that in the CLS sample. This can increase compliance with the legislation that establishes a maximum phosphorus content of 0.4% for starches cross-linked with the STPP/STMP mixture (USDA, 2019). The ECLS sample had phosphorus content within the established legislation, which is an advantage when compared with the CLS sample. After modification through the conventional process, for the modified starch to have phosphorus content within the established legislation, several washes are necessary to remove excess phosphorus, thus leading to excessive water consumption, effluent production, and longer preparation time. Therefore, the dual modification process to which the ECLS sample was subjected could be more beneficial than the processing method used on the CLS sample since it was faster, yielded proper cross-linking, and did not exceed the phosphorus established legislation even when the two samples were prepared with the same content of reagents.

Lemos *et al.* (2020) carried out the modification of potato, banana, corn, cassava, and breadfruit starches with STMP and STPP through the conventional method by heating in an oven with the washing of excess phosphorus, thereby obtaining phosphorus contents for modified potato, banana, corn, cassava, and breadfruit starches of 0.084, 0.066, 0.037, 0.054, and 0.050, respectively, and degrees of substitution of 0.004, 0.003, 0.001, 0.002, and 0.002, respectively. The values presented by Lemos *et al.* (2020) for the phosphorus content and degree of substitution were lower than those observed in the present work. The repeated washings of cross-linked starch can explain this removal of excess phosphorus. Washing can efficiently keep the phosphorus content within the limits established by legislation. However, it can remove more phosphorus than necessary, thus decreasing the modification efficiency observed in the results for phosphorus content and degree of substitution. The phosphorus content makes it possible to assess the degree of cross-linking indirectly. The greater the amount of phosphorus, the greater the cross-linking performed. In the cross-

linking reaction with STMP and STPP, the hydrophilic groups of the phosphorus react with the hydroxyls of the starch chains, thus producing di-starch phosphate. The more STMP is used in the reaction, the more phosphate groups are incorporated into the starch, and the more they are added to the granule inside, which can be observed indirectly by the degree of substitution (Lemos *et al.*, 2020).

Relating the phosphorus content with the amylose content (Table 1) present in the samples, it was observed that the amylose content decreased as the phosphorus content increased. O'Brien and Wang (2009) also made this observation in their corn starch samples prepared at pH 9.0. Landerito and Wang (2005b) described that amylopectin has a branched structure that could retain more phosphates in the crystalline region for phosphorylation; thus, when the amylose content is lower, the amylopectin content is higher, and thus the content of phosphorus also increases, which can explain the lower amylose content in cross-linked samples.

Paste clarity

Based on the results of the clarity analysis shown in Figure 2, the CLS sample was the one with the highest transmittance between the samples, and there was no significant difference between the NS and ECLS samples. Based on the results, it was also estimated that the conventional modification to which the CLS sample was subjected produced pastes with greater transparency when compared with the dual modification used on the ECLS sample. The ENS sample showed a transmittance of less than 40%, the lowest value among all samples. A lower transmittance indicates more opaque starch paste.
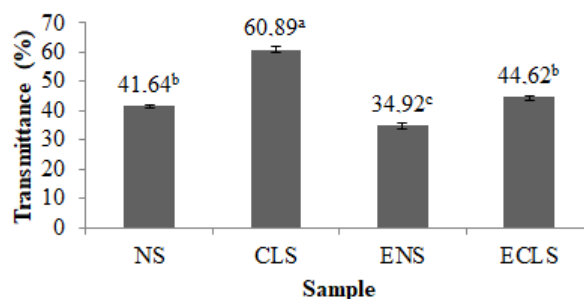

Figure 2. Paste clarity results. Similar lowercase superscripts indicate no statistical difference between the samples.

Starch transparency data obtained by clarity analysis is important for developing starch-based products, as this information can be used to make choices of starch processing and application methods. Starch transparency can be affected by several factors such as reflection and refraction of light, species, colour, and granular structure of the starch, and groups of starch (Chen *et al.*, 2017).

Swelling and solubility

Based on the swelling power values of the samples shown in Table 1, there were no significant differences between the CLS and ECLS samples, and they had greater swelling capacities at 90°C when compared with the other samples. The ENS sample was the one with the lowest swelling capacity.

The greater swelling capacity of the CLS and ECLS samples might have been due to the insertion of negatively charged phosphate groups that decreased the hydrogen bonding capacity between nearby molecules, which permitted water penetration and increased swelling, as well as amylose gel formation. The addition of hydrophilic phosphorus groups by covalent phosphate diester bonding increased the water absorption capacity. This occurred due to the repulsion of phosphate groups, which reduced the strength of the intermolecular bonds between the starch chains, where more water molecules can be accommodated (Lemos *et al.*, 2020). The lower swelling power of the ENS sample can be explained by the fragmentation of the starch granules, as starch gelatinisation occurred during the extrusion process, thus decreasing water absorption and granule swelling (O'Brien and Wang, 2009; Sharma *et al.*, 2015).

The samples that were modified through the extrusion process showed the highest solubility, and the ECLS sample that underwent double modification had the highest solubility among all samples. Sharma *et al.* (2015) also observed that extruded starches had greater solubility than native starches or those that had undergone the conventional modification process. They studied the effects of extrusion on corn, pea, and red bean starches, and found that extruded starches had solubilities between 7.63 and 12.57%, and their counterparts 0.71 to 5.64%, which could be associated with starch degradation during extrusion, thus generating a greater amount of free soluble polysaccharides which might have occurred due to the leaching of amylose from the starch granules, and

the high temperature of the extrusion process and shear force (Shi *et al.*, 2013; Sharma *et al.*, 2015).

Gelatinisation degree

In addition to greater solubility, extruded samples also showed a higher degree of gelatinisation, with values of 73.26 and 89.85% for ENS and ECLS, respectively, as shown in Figure 3. Starches modified by extrusion are already considered pre-gelatinised, and can absorb water rapidly upon exposure, thus increasing the plasticising effect of water and facilitating gelatinisation of starch modified by extrusion (Juansang *et al.*, 2012). This is an advantage when these starches are applied to technological processes, where no heat is applied, and gelatinisation is required. Pregelatinised starches have been applied mainly as thickeners in baby foods, soups, and instant desserts (Ashogbon and Akintayo, 2014).
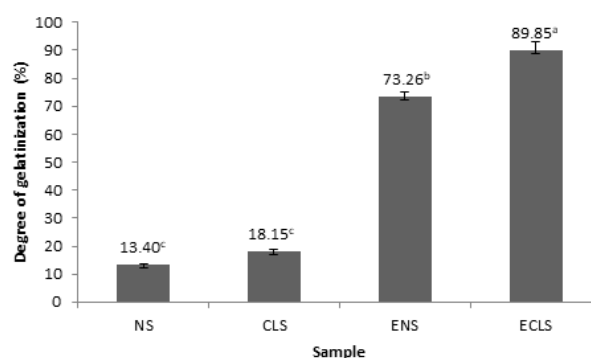
Figure 3. Gelatinisation degree. Similar lowercase superscripts indicate no statistical difference between the samples.

On the effect of gelatinisation of modified sugarcane starch as described by Juansang *et al.* (2012), the authors explained that the extent of disintegration of starch granules can be indicated by the degree of gelatinisation. A higher gelatinisation degree indicates a greater extent of dispersion of the starch granules. The greater gelatinisation degree of the ECLS sample can be correlated with its higher swelling power when compared with the NS and ENS samples. These facts, added to the phosphorus content of the samples, can be attributed to the substitution groups that can facilitate the entry and absorption of water into the starch granules by breaking the hydrogen bonds, thus increasing the space between the starch chains due to the hydrophilic character of the substituent (Juansang *et al.*, 2012).

Pasting properties

Table 2 presents the results of the analysis of pasting properties, and Figure 3 presents the behaviour of the samples during the analysis. The pasting temperature of the extruded samples did not differ significantly, and the NS sample had a higher pasting temperature. Pasting temperature indicates the beginning of starch gelatinisation, and is shown in Figure 3, where the formation of the curve begins.

Table 2. Pasting properties of starches.

Sample	Pasting temperature (°C)	Peak viscosity (cP)	Trough (cP)	Breakdown viscosity (cP)	Final viscosity (cP)	Setback (cP)
NS	85.45[a]	1,685.00[a]	1,062.00[a]	623.00[a]	1,759.00[a]	697.00[a]
CLS	79.23[b]	1,358.00[b]	1,099.00[a]	259.50[c]	1,413.00[b]	314.00[b]
ENS	47.95[c]	395.00[c]	0.00[c]	395.00[b]	12.00[c]	12.00[c]
ECLS	47.95[c]	440.50[c]	0.00[c]	440.50[b]	0.00[c]	0.00[c]

Similar lowercase superscripts indicate no statistical difference between the samples based on Tukey's test ($p > 0.05$).

The extruded starches had a low pasting temperature as they were already pre-gelatinised, thus, when starting the analysis at temperatures below 50°C, the formation of the curve and the viscosity peak were observed. As the temperature increased during the analysis, the gel structure was broken, and the viscosity was practically zero. Landerito and Wang (2005a) also made this observation when evaluating changes in different types of corn starch through reactive extrusion, where extruded starches showed a lower viscosity profile when compared with native starches or starches modified by conventional processes, and attributed this fact to the disintegration of the starch structure by the characteristics of the high-shear, -pressure, and -temperature extrusion process. When the starch granule is damaged, water can penetrate more easily, consequently lowering the gelatinisation temperature, and producing a lower viscosity profile at high temperatures. The low pasting temperature of extruded starches indicated that they could increase in volume in cold water, thus increasing the viscosity of aqueous systems that are not heated quickly. With this characteristic, this type of starch is useful in food production processes where there is no heat available, when the formulation requires thickening before heating, when in none of the production steps sufficient heat is used to cook the starch, or when heat cannot be applied due to the thermal instability of another ingredient, for example, in the production of gum (BeMiller, 2016). In research by Li *et al*. (2020), the addition of pregelatinised corn starch improved the quality of processing and noodle intake when compared with native corn starch.

The NS sample showed the highest viscosity peak. Peak viscosity is the maximum viscosity achieved during heating. Native corn starches tend to have high viscosity with increasing temperature and agitation. The CLS sample showed lower peak viscosity values as compared to the NS sample. However, the ECLS sample showed an even lower viscosity peak than the CLS sample. Cross-linked starches showed a low viscosity peak as a result of the formation of a diphosphate bond in their structures, when doubly modified in combination with the extrusion process, the peak of viscosity tried to decrease even more. A lower viscosity peak can provide improved thermal granular stability and soft gels, and starch with these characteristics can be applied for use in frozen foods, weaning baby foods, and soft confectionery products (Ashogbon, 2021).

The viscosity results demonstrated that the extruded samples did not present this value. The trough is the minimum viscosity achieved after a constant temperature of 95°C. These samples did not show viscosity at temperatures above 50°C; therefore, they did not show trough.

Breakage values ranged between 623 and 259.50 cP, with the highest value being for NS starch and the lowest for CLS. The trough was calculated from the difference between the peak and minimum viscosity at 95°C. The extruded samples had minimal final viscosity; when the temperature of the analysis was lowered, these starches began to show viscosity again. As for the NS and CLS samples, the final viscosity was higher than the peak viscosity, which is a common characteristic of native corn starch. Final viscosity indicates the starch's ability to form a

viscous paste; it is the maximum viscosity value reached at a cooling temperature of 50°C.

The setback measures the syneresis after cooling of a starch paste that has been cooked to 95°C; it is measured by the difference between the final viscosity at 50°C and the minimum viscosity at 95°C. Extruded starches are less susceptible to syneresis as can be seen from the results obtained from the setback. Furthermore, the setback decreased in cross-linked starches; this trend is linked to the decrease in the amorphous mobility of the starch granule chains attributed to intermolecular bonds. This justifies the CLS sample (314 cP) presenting a lower setback than the NS sample (697 cP). Setback occurs when starch molecules attempt to return to their original structure, thus causing syneresis, which is the expulsion of the water that forms the gel with consequent reconstitution of the intermolecular interactions between the starch molecules, which is often a problem for industrial processes; therefore, a low syneresis as shown by the ECLS sample can be advantageous in food production.

From Figure 3, the difference in the behaviour of the curves of the extruded samples as compared to the NS and CLS samples is clear. Shi *et al.* (2013) observed the same behaviour in the viscosity curves of their cross-linked starch samples; their curves were almost a straight line as compared to those of native starch, and they attributed this fact to the strength of the additional covalent bonds of the cross-linked starch, which allowed the granules to resist the gelatinisation temperature (Juansang *et al.*, 2012).

Conclusion

Reactive extrusion as a dual modification to obtain cross-linked corn starch using a single-screw extruder could be a promising alternative to conventional modification. Reactive extrusion was able to perform the cross-linking reaction, inserting phosphate groups in the starch chain, and providing a modified starch with specific functional properties. Cross-linked starch modified through reactive extrusion was quick to prepare, did not require large volumes of water, and did not generate effluents. Starch cross-linked by reactive extrusion had phosphorus content within the limits established by legislation without the need to wash out excess reagents, in addition to having characteristics of swelling, solubility, gelatinisation, and pasting properties that favour their application in various food products, especially those that do not require the application of high temperatures for their preparation such as instant foods. Further studies are needed on the applications of cross-linked starch produced by reactive extrusion.

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