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Optimizing the Production of Phosphated Distarch Phosphate (E1413) and Evaluating its Physicochemical Properties in Stirred Yogurt

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Abstract

The objective of this work was to optimize the production of phosphated distarch phosphate (E1413) from potato starch using two phosphorylation methods-mono/disodium phosphate and sodium tripolyphosphate (STPP)-followed by crosslinking with sodium trimetaphosphate (STMP). Optimization involved varying phosphate concentration (5-50%), mono/disodium phosphate ratio (1:1 to 3:1) and heating time (1-3 h) at 130 °C to maximize phosphorus content and degree of substitution. After crosslinking the optimized starches by STMP, they were evaluated for transparency, swelling power, viscosity, and thermal properties. In addition, the functional performance of these modified starches was evaluated in stirred yogurt, focusing on parameters such as syneresis, viscosity, and texture hardness. The results showed that the degree of phosphorylation and substitution were significantly influenced by the phosphate concentration and the mono/disodium phosphate ratio, with the optimal conditions being (1) a 5% phosphate compound with a mono/diphosphate ratio of 2.9 and a time of 1 h, which was then crosslinked with 2% STMP, and (2) a mixture of 2% STMP and 5% STPP at an optimal pH of 9.5 (STMP-STPP). The incorporation of E1413 modified starch into stirred yogurt enhanced its physicochemical properties, notably by increasing viscosity and reducing syneresis. Comparing the hardness of the modified starch samples, it is clear that the E1413 sample at 2% had the highest hardness, which was consistent with the viscosity results. The study concluded that carefully controlled dual modification of potato starch produces functional E1413 starches suitable as effective stabilizers in dairy applications, improving yogurt consistency and shelf life.

Keywords
Crosslinking
RVA
Starch phosphate
Stirred yogurt
Viscosity

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Introduction

Native starch, extracted directly from plants, has limited industrial applicability due to its low solubility and poor resistance to heat, acidity, and mechanical stress. These limitations hinder its direct use in food processing and other manufacturing environments. To address these challenges, starch is modified through physical, chemical, or enzymatic methods to enhance its functionality. Modified starches exhibit improved stability, viscosity, and texture, making them suitable for diverse applications across the food, pharmaceutical, textile, and packaging industries. Among these, dual modifications, such as crosslinking and phosphorylation, have shown particular promise in improving thermal stability and reducing syneresis in dairy products like yogurt (Bertolini, 2009).

Phosphorus oxychloride and three inorganic phosphate salts (sodium orthophosphate, sodium tripolyphosphate (STPP), and sodium trimetaphosphate (STMP)) are used to prepare food starch phosphate. Starch phosphate refers to a phosphate monoester in which one of the hydroxyl groups of the starch molecule is esterified with phosphoric acid, resulting in the formation of a starch monoester. The permissible phosphorus content in these products is 0.5% for wheat and potato starch and 0.4% for starch from other sources (Wurzburg *et al.*, 1980). The granule size of modified starch tends to increase with a higher degree of substitution, as chemical modifications alter the molecular structure and packing density of starch polymers (Abdul Hadi *et al.*, 2020). This increase may be due to the degree of substitution of phosphate groups within the modified granules, which creates specific repulsive forces that increase the size

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of the starch granules. Starch phosphate has a transparent paste with high consistency, good freeze-thaw stability, and good emulsifying properties (Singh *et al.*, 2009).

Phosphate salts can cause crosslinking between linear or branched amylopectin chains. These modified starches form ether or ester bonds with the hydroxyl groups of starch molecules (Singh *et al.*, 2016). In this class of starches, granule strength is enhanced through the formation of a chemically cross-linked three-dimensional network, which reinforces the internal structure and improves resistance to mechanical, thermal, and acidic conditions (Woo & Seib, 2002). Crosslinking reinforces the structural integrity of swollen cooked starch granules, thereby minimizing granule disruption and reducing viscosity breakdown under acidic and shear conditions. This type of starch exhibits enhanced stability under freeze-thaw conditions. It shows a lower retrogradation rate and a higher gelatinization temperature. These properties result from crosslinking, which introduces covalent bonds between starch molecules, restricting them (Mali *et al.*, 2001).

A single chemical modification, particularly crosslinking, can induce undesirable characteristics in starch-such as reduced swelling power and an increased tendency for retrogradation (Kurakake *et al.*, 2009). The adverse effects of crosslinking (such as reduced paste transparency and stability at cold temperatures) are also improved by modifications such as esterification or etherification. Therefore, double modification is a widely used method to tailor starch properties for specific food applications. It typically results in starches with improved structural strength and higher viscosity compared to their native starches. However, the effectiveness of dual modification of starch largely depends on the specific processing method employed (Singh *et al.*, 2016). The production of distarch phosphate is a type of chemical double modification, which is usually carried out in the presence of STMP and STPP and in the pH range of about 9.5-9 (Ali *et al.*, 2024; Lim & Seib, 1993). Here, the replacement of the phosphate group of STPP can overcome some of the drawbacks of crosslinking with STMP (Sang *et al.*, 2010).

During phosphorylation, pH significantly determines the ratio of monoester bonds to diester bonds (Kerr & Cleveland, 1960; Nierle, 1969). The efficiency of starch phosphorylation varies depending on the starch type and the modification method. For example, dry phosphorylation using sodium phosphates achieves optimal results at 160 °C for 3 h under pH 6 conditions, yielding a higher degree of substitution in amylose than in amylopectin (Sitohy *et al.*, 2000). In a half-dry process utilizing cassava starch, optimal conditions consist of a reaction temperature of 150 °C, a reaction duration of 1 h, and a pH of 5, resulting in a phosphorus content of 1.190%. Furthermore, cassava starch phosphorylated at pH 9.0 using sodium phosphates exhibited significant changes in pasting behavior, including reduced peak viscosity, increased pasting temperature, and limited breakdown during heating. These alterations, along with changes in digestibility, suggest that increased phosphorus content can disrupt granule morphology and restrict swelling, ultimately decreasing overall viscosity (Zhongdong *et al.*, 2008).

Zhang *et al.* (2017) produced edible corn starch phosphate by reacting starch with STPP under optimized conditions. Key findings showed that optimal starch phosphate was achieved at 0.39% phosphate with a reaction time of 90 min, temperature of 160 °C, and pH of 5.0. Improved freeze-thaw stability and paste transparency were noted compared to native starch, with significant differences in syneresis and paste characteristics ($P<0.05$). Muhammad *et al.* (2000) investigated the effect of pH on the phosphorylation of wheat and corn starch using STPP and STMP alone and in combination. They observed that increasing the pH (from 8 to 10) when using STMP increased the phosphorus content of the samples. However, increasing the pH from 6 to 10 when using a mixture of STMP and STPP decreased the phosphorus content of the samples. Lim and Seib (1993) also found that wheat and corn starch phosphates prepared at pH 5.9 with a mixture of phosphate salts gave viscoelastic pastes compared to pastes prepared with STPP alone at pH 10 for wheat and pH 11 for corn.

In the food industry, low-substituted starches, those with minimal chemical modification, are used as stiffening agents, emulsifiers and stabilizers in various products. High-phosphate-substituted and cross-linked starches are products with high swelling power that can be used as water absorbents (hydrogels and super absorbents) in cosmetic, pharmaceutical and agricultural products (Passauer *et al.*, 2010). The degree of substitution of phosphate groups strongly affects the properties of starch phosphate (such as solubility, swelling power, rheological behavior, syneresis, and retrogradation), (BeMiller & Whistler, 2009). This modified starch is commonly used as a stabilizer in yogurt, especially stirred yogurt, to provide desirable textural properties and prevent syneresis (Pang *et al.*, 2019).

This study was conducted to optimize the synthesis of monostarch phosphate (E1410) using sodium mono- and diphosphates. Subsequently, phosphated distarch phosphate (E1413) was produced through crosslinking with sodium trimetaphosphate (STMP) and sodium tripolyphosphate (STPP). The resulting dual-modified starches were evaluated for their physicochemical properties, including viscosity, gel strength, and thermal stability. Finally, the most promising E1413 sample was incorporated into stirred yogurt to assess its functional performance in terms of reducing syneresis, enhancing viscosity, and improving texture.

Materials and methods

Materials

Mono- and disodium phosphate, sodium hydroxide, hydrochloric acid, sodium sulfate, acetone, and ethanol were purchased from Dr. Mojallali Company (Tehran, Iran). STPP, STMP, ammonium molybdate, and ammonium vanadate were purchased from Merck Company (Darmstadt, Germany). Potato starch was obtained from Talachin Company (Abyek-Qazvin, Iran). Low-fat milk (1.5% fat) and yogurt starter culture were purchased from Pegah Company (Tehran, Iran).

Optimization of starch monophosphate (E1410) production

Two methods can produce starch monophosphate: mono/disodium phosphate or STPP. In this study, both methods were investigated.

Optimization of starch monophosphate production using mono/disodium phosphate

The mono-phosphorylation of starch was performed by reacting starch with a mixture of sodium dihydrogen phosphate monohydrate and disodium hydrogen phosphate dihydrate in a semi-dry process. The phosphate salts were first dissolved in water at 35 °C with pH adjusted to 5 using sodium hydroxide, then mixed with starch and stirred at room temperature. The resulting slurry was filtered, dried at 55 °C for 24 h, homogenized, and further dried at 65 °C before phosphorylation at 150 °C for 3 h. After cooling, the product underwent purification through washing with aqueous methanol to remove unreacted phosphates, followed by dehydration with ethanol. The purified starch phosphate was then suspended in water, precipitated with acetone, washed with ethanol, filtered, and finally dried at 45 °C to obtain the final product, according to the method of Passauer *et al.* (2010). According to Table (1), the test variables included the phosphate concentration (5-50% w/w starch), the ratio of monosodium dihydrogen phosphate to disodium hydrogen phosphate (1:1 to 3:1), and the heating time (1-3 h) at 130 °C.

Crosslinking of starch (E1412)

Crosslinking of potato starch was carried out according to the method of Shi *et al.* (2013). First, 50 g of potato starch (or starch phosphate) was placed into a beaker, and 2% (w/w, based on starch dry weight) of the crosslinking agent (STMP (99%)/STPP (1%)) was added. Subsequently, 70 mL of distilled water was added, and the pH was adjusted to 11 using 0.1 N sodium hydroxide (NaOH) solution, chosen to optimize STMP crosslinking efficiency, which requires alkaline conditions above pH 9. The mixture was stirred at 45 °C for 3 h on a magnetic stirrer, with intermittent pH adjustment to maintain stability. Neutralization was performed using 0.1 N hydrochloric acid (HCl) solution to reach pH 6. The product was separated by vacuum filtration (EPEC, China) after each of the four washing cycles with distilled water and subsequently dried in a laboratory oven at 40 °C for 24 h (Shi *et al.*, 2013).

Optimization of the production of phosphated distarch phosphate (E1413) using STPP and STMP

5 g STPP (equivalent to 5% w/w of starch) and 2 g STMP (equivalent to 2% w/w) were dissolved in 300 mL of water containing 5 g sodium sulfate. The pH of the solution was adjusted between 9.5 and 11 by adding 3% (w/v) sodium hydroxide to ensure crosslinking. 100 g of starch was dispersed in the prepared solution, the mixture's pH was readjusted, and the total weight was reached by adding water to about 450 g. The dried starch cake was placed in a Petri dish and subjected to heat treatment in a laboratory oven at 130 °C for 2 h to induce phosphorylation. it was performed under dry conditions to facilitate phosphate group formation within the starch matrix. After cooling to room temperature, the phosphorylated starch was dispersed in approximately 200 mL of distilled water. The suspension was adjusted to pH 6.5 using either 0.1 N hydrochloric acid (HCl) or 0.1 N sodium hydroxide (NaOH) as needed. The starch was washed three times with 200 mL of distilled water per cycle, using vacuum filtration for solid-liquid separation, and finally dried in an oven (Shimaz, Iran) at 40 °C (Kerr & Cleveland, 1959).

Phosphorus content and degree of substitution measurement

Due to the naturally low phosphorus levels in potato starch, the phosphorus content was quantified using a modified spectrophotometric method with ammonium molybdate and ammonium vanadate reagents to enhance sensitivity, as described by (Zhao *et al.*, 2012). Briefly, 50 mg of the starch sample was digested with concentrated nitric acid and hydrogen peroxide, followed by dilution. A mixed reagent containing ammonium molybdate and vanadate was added, and the absorbance was measured at 400 nm by spectrophotometer (Biochrom, England). The phosphorus concentration was then calculated using a standard calibration curve. The degree of substitution (DS), defined as the average number of phosphate groups per glucose unit, was calculated using the following Eq. (1):

$$DS_p = \frac{162P\%}{3100 - 103P\%} \quad (1)$$

Where P% is the phosphorus content expressed as a percentage, and the constants account for molecular weights of glucose and phosphate.

Determination of past transparency (light transmission)

Paste transparency was determined using the method of Sitohy *et al.* (2000) by measuring the percentage transmission (T%) of a 1% (w/w) starch solution (pH 6.5) at 650 nm after boiling in a water bath for 30 min. The measurements were performed using a UV-Visible spectrophotometer (UV-1800, Shimadzu Corporation, Kyoto, Japan).

Determination of solubility and swelling power

A 2% suspension of starches was prepared and heated in a hot water bath at 95 °C for 30 min with stirring. After cooling the samples to 25 °C, they were centrifuged at 2500 g for 15 min. The dry matter of the supernatant solution and the resulting gel were obtained by drying them in an oven at 105 °C to obtain starch's solubility and swelling power using the following relationships (Ačkar *et al.*, 2010).

$$\text{WSI} = \frac{W1}{0.1} * 100\% \quad (2)$$

$$\text{SP} = \frac{Ws}{0.1\ (100\% - WSI)} \quad (3)$$

Where WSI is the water solubility index, W_1 is the weight of the dried supernatant, and W_s is the weight of the precipitate.

Pasting characteristics analysis using rapid visco analyzer (RVA)

The pasting properties were evaluated using a rapid visco analyzer (RVA; Starch Master 2, Perten Instruments, Australia) following Zhang *et al.* (2020), method where 3.0 g of each starch sample was suspended in 25.0 g distilled water (10.7% w/w solids concentration) in an aluminum canister. The standard temperature profile included equilibration at 50 °C, heating to 95 °C, a holding phase, cooling back to 50 °C,

and a stabilization period. Key pasting parameters-peak viscosity (PV, the maximum viscosity during heating), final viscosity (FV, viscosity after cooling), breakdown (BD, PV minus trough viscosity, indicating thermal stability), and setback (SB, FV minus trough viscosity, reflecting retrogradation tendency), were recorded in centipoise (cP) with triplicate measurements under controlled conditions to ensure reproducibility (Zhang *et al.*, 2020).

Determination of yogurt syneresis (SI)

Syneresis refers to the separation of serum from the yogurt gel matrix, and it is considered an important indicator of gel stability and water-holding capacity. For yogurt production, Low-fat milk (1.5% fat) was heated to 85 °C and held for 20 min, then cooled to 43 °C. The selected modified starch (E1413) was added at 1% (w/w) concentration and mixed thoroughly. The yogurt starter culture (*Streptococcus thermophilus* and *Lactobacillus delbrueckii* subsp. bulgaricus) was inoculated at 0.02% (w/w), and the mixture was incubated at 43 °C until the pH reached 4.6. After fermentation, the yogurt was cooled to 4 °C and stored for 24 h before syneresis analysis. Syneresis was determined by centrifuging (Sigma, Germany) 50 g of yogurt at 5000 × g for 10 min at 4 °C, and the released whey was measured and expressed as a percentage of the total weight. The syneresis index was expressed as grams of liquid separated per 100 g of yogurt (g/100 g) using Eq. (4), (Amaya-Llano *et al.*, 2008).

$$SI = \frac{L}{W} \times 100 \quad (4)$$

Where SI is the syneresis index, the L is the weight of the separated liquid, and the W is the initial weight of the sample (Raftani Amiri *et al.*, 2024).

Measurement of yogurt viscosity

The viscosity of yogurt, which indicates the resistance to flow, was measured using a rheometer (Anton Paar MCR302, Graz, Austri) with a cone-plate geometry of 50 mm in diameter, a cone angle of 1°, and a gap of 0.05 mm. About 4 g of the sample was weighed accurately, placed in the measurement system, and left for 10 min at 4 °C to regenerate its structure. The viscosity of the samples was evaluated based on shear rate scanning. The yogurt flow curves at 20 °C were obtained by varying the shear rate from 0.03 to 200 s^{-1} (Abbas *et al.*, 2017).

Yogurt texture

Hardness of samples was measured using an STM1 Texture Analyzer (Santam, Tehran, Iran), equipped with a 5-kg load cell. A cylindrical stainless-steel probe (3 cm diameter; supplied by Santam, Tehran, Iran) penetrated the samples by 10 mm, and the force corresponding to this penetration depth was recorded. During testing, samples were placed in cylindrical containers with an internal diameter of 4 cm and a height of 5 cm to ensure consistent positioning and support (Saleh *et al.*, 2020).

Statistical analysis

To optimize the production of starch monophosphate (E1410) using mono/disodium phosphate, response surface methodology (RSM) was employed in the form of a central composite design (CCD), along with analysis of variance (ANOVA), using Design Expert 7 statistical software. The center point was tested in three replicates to estimate experimental error. For each factor, the test range was selected based on preliminary trial results. Following optimization, confirmatory experiments were conducted under optimal conditions, and the average observed values were compared with those predicted by the model. To assess differences between starch monophosphate samples produced by the STPP method and those from the optimized process, variance analysis was performed using SPSS 21 software and Duncan's multiple range test at a 95% confidence level. All reported values are based on a minimum of two replicates.

Results and discussion

Optimization of starch phosphate production using mono- and disodium phosphate

The phosphorus values of the samples under different test conditions are shown in Table (1). The variance analysis (ANOVA) results of multiple regression models for phosphorus content and degree of substitution of starch phosphate were examined. The analysis revealed that the quadratic model provided an excellent fit for predicting the phosphorus content in the starches. The model demonstrated a high coefficient of determination ($R^2 = 0.995$), indicating that 99.5% of the variability in the response was accurately accounted for by the model. Also, the adjusted coefficient of variation ($R^2_{adj} = 0.989$) is close to the coefficient of variation, indicating a high correlation between the test and predicted values.

Table 1. Variables and levels used in the central composite method to assess phosphorus levels and the degree of replacement

Standard No.	Phosphate Con. (g/100 g starch)	Mono/Di phosphate Ratio	Time of heating (h)	P (%)	DS_p (%)
1	5	1	1	0.36	0.019
2	50	1	1	2.07	0.116
3	5	3	1	0.39	0.021
4	50	3	1	2.43	0.138
5	5	1	3	0.38	0.020
6	50	1	3	2.06	0.115
7	5	3	3	0.39	0.021
8	50	3	3	2.64	0.151
9	5	2	2	0.45	0.024
10	50	2	2	2.67	0.153
11	27.5	1	2	1.26	0.069
12	27.5	3	2	1.57	0.086
13	27.5	2	1	1.30	0.071
14	27.5	2	3	1.29	0.071
15	27.5	2	2	1.48	0.081
16	27.5	2	2	1.47	0.081
17	27.5	2	2	1.44	0.079

P (%): Phosphorus content as percentage of starch dry weight, DS_p (%): Degree of substitution with phosphate groups.

The lack-of-fit test assesses whether the model appropriately represents data points outside the regression range. As shown in Table (2), the lack-of-fit was statistically insignificant, suggesting that the model is suitable. Overall, the results confirmed that the quadratic regression model reliably predicts the phosphorus content of starches across

varying conditions of phosphate concentration, mono-to-disodium phosphate ratio, and heating time.

Table 2. Examination of the coefficients of the quadratic model for assessing phosphorus levels

Source	Sum of squares	df	Mean square	Value	Prob > *F*	
Model	10.16	9	1.13	167.87	< 0.0001	significant
A-Con	9.77	1	9.77	1452.42	< 0.0001	
B-Mo/Di	0.17	1	0.17	24.92	0.0016	
C-time	4.88E-03	1	4.88E-03	0.73	0.4225	
AB	0.1	1	0.1	15.18	0.0059	
AC	4.05E-03	1	4.05E-03	0.6	0.4632	
BC	5.09E-03	1	5.09E-03	0.76	0.4131	
A^2	0.023	1	0.023	3.42	0.1071	
B^2	6.98E-03	1	6.98E-03	1.04	0.3423	
C^2	0.073	1	0.073	10.86	0.0132	
Residual	0.047	7	6.73E-03			
Lack of fit	0.046	5	9.22E-03	18.3	0.0526	not significant
Pure error	1.01E-03	2	5.04E-04			
Total	10.21	16				

A quadratic multiple regression analysis was conducted on the experimental data, resulting in Eq. (5), which expresses the predicted values for total phosphorus content in numerical form.

(5)

$$\text{Phosphorus content (\%)} = -0.34 + 0.022\ A + 0.145\ B + 0.60\ C + 0.005\ AB + 0.001\ AC + 0.025\ BC + 1.83\ A^2 - 0.051\ B^2 - 0.165\ C^2$$

In this relation, A, B and C are the codes for the amount of phosphate compounds, the ratio of mono to disodium phosphate and the heating time, respectively.

As can be seen in Table (2), the linear effect of variables A and B and the interaction effect of AB significantly affected the phosphorus content of starches ($P < 0.05$), so that an increase in both variables, the amount of phosphate compounds and the ratio of mono to disodium phosphate, increased the phosphorus content of the samples. Therefore, the monophosphate factor is more effective than diphosphate in creating phosphate groups in starch. As shown in Fig. (1), an increase in the amount of phosphate compounds at higher ratios of mono to diphosphate causes a greater increase in the phosphorus content of modified starches, which indicates the interaction effect of these two indicators.

Passauer *et al.* (2010) produced starch phosphate using a mixture of monosodium dihydrogen monohydrate and disodium hydrogen phosphate at molar ratios of phosphate compound to starch ranging from 0.15 to 1.63 and molar ratios of monosodium hydrogen phosphate to disodium hydrogen phosphate ranging from 1.3 to 3.8. Their results showed that the degree of starch substitution ranged from 0.02 to 0.37 (equivalent to 0.375 and 5.75% phosphorus), depending on the amount of phosphate compound used (Passauer *et al.*, 2010).

As mentioned, the permissible phosphorus content in food-grade modified potato starch is 0.5%. On the other hand, increasing phosphate groups can impart positive properties to starch, such as improved paste clarity and viscosity, enhanced freeze-thaw stability, superior emulsifying capacity, greater shear and acid resistance, and improved water retention. Therefore, the phosphorus content was maintained below 0.5% (or a degree of substitution of 0.0266) to ensure both functional benefits and regulatory compliance.

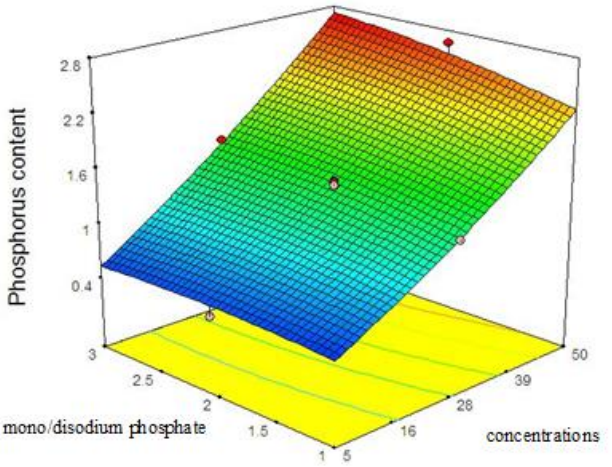

Fig. 1. Response surface diagram of the combined effect of mono/disodium phosphate (Mo/Di) and their concentrations (Con) on the phosphorus content of E1410 starches.

Through systematic optimization, it was determined that the most favorable conditions for phosphate compound processing involve maintaining a 5% phosphate compound concentration with a mono-to-diphosphate ratio of 2.9 and a reaction time of 1 h. These parameters yielded a desirability score of 1, confirming their optimal nature. Under these conditions, the predicted phosphorus content was calculated to be approximately 0.4%, which showed excellent agreement with the experimentally measured value of 0.39%. This close correlation between predicted and actual results demonstrates the high reliability of the model, with a predictive accuracy of 97.5%. The findings highlight the effectiveness of the optimization approach in achieving precise control over phosphate compound characteristics.

Production of phosphated distarch phosphate (E1413) using STMP

According to Table (3), the starch cross-linked with STMP at a pH of 11 had 0.13% phosphorus and a degree of substitution of 0.014%. The maximum phosphorus level accepted by the US federal regulations when using STMP is 0.4%, and since the phosphorus content of raw starch was about 0.08%, the produced starch is suitable for use in food products.

Starch was phosphorylated using a mixture containing 2% STMP and 5% STPP in the presence of 5% sodium sulfate at pH values of 9.5 and 11. Under these conditions, the total phosphorus content of the modified starch ranged from 0.23 to 0.27%, showing no significant variation. Similarly, Muhammad *et al.* (2000) modified sago starch in a semi-dry state using 2% STMP combined with 5% STPP, applying pH levels ranging from 6 to 11. Initially, as the pH increased from 6 to 8, the phosphorus level gradually decreased, but at pH 8 and above, the phosphorylation increased rapidly, reaching a maximum at pH 10 (0.119%). They reported that the optimal pH for phosphorylation, regarding phosphorus content, is between 9 and 9.5. Lim and Seib (1993) stated that with a mixture of 2% STMP and 5% STPP in the presence of 5% sodium sulfate, an initial pH higher than 9.5 is not recommended for the phosphorylation of wheat and corn

starches due to the extreme sensitivity of the crosslinking reaction at pH > 9.5, and a reaction pH of 9.5-9 seems reasonable.

According to Lim and Seib (1993), when phosphorylating wheat and corn starches with a mixture containing 2% STMP and 5% STPP in the presence of 5% sodium sulfate, an initial reaction pH exceeding 9.5 is not recommended due to the pronounced sensitivity of the crosslinking process at higher alkalinity. A reaction pH in the range of 9.0-9.5 is considered optimal. Within this pH range, starches treated under these conditions typically exhibit a phosphorus content of approximately 0.4%. Consequently, a reaction pH of 9.5 was selected for the production of E1413, as it facilitates both phosphorylation and crosslinking processes effectively.

Table 3. Effect of pH and phosphatizing compound on phosphorus content and degree of replacement of phosphorylated starches with STPP and STMP combination

Different STPP Con.	Phosphorus content (%)	DS_p (%)
STMP (2%) pH = 11	0.13 ± 0.02^{c}	0.007 ± 0.001^{c}
STMP (2%) + STPP (5%) pH = 9.5	0.27 ± 0.03^{b}	0.014 ± 0.003^{b}
STMP (2%) + STPP (5%) pH = 11	0.23 ± 0.03^{b}	0.012 ± 0.002^{b}
M.D (5%) + STMP (2%) pH = 9.5	0.41 ± 0.03^{a}	0.022 ± 0.002^{a}

Data are represented as mean values ± SE (n = 3). Within the same column, means with different superscripts are significantly different ($P < 0.05$). STMP: Sodium orthophosphate, sodium tripolyphosphate, STPP: Sodium trimetaphosphate, and M.D.: Mono/Di phosphate Ratio.

Also, considering that the aim of this research was to produce dual starches, dual phosphorylated samples including (1) a mixture of 2% STMP and 5% STPP at an optimal pH of 9.5 (STMP-STPP), (2) a sample phosphorylated with 5% phosphate compound with a mono-to-diphosphate ratio of 2.9: 1 and a time of 1 hour, which was then crosslinked with 2% STMP (M.D-STMP), were selected for physicochemical and rheological evaluations.

Transparency of the paste of optimized E1413

As shown in Fig. (2), the highest transparency among the dual phosphorylated starches was observed in the M.D-STMP sample, which incorporated both mono- and di-phosphates before crosslinking.

Fig. (2), shows that the light transmittance of native potato starch (control sample) is approximately 32%, which is significantly lower than the 96% reported by Lim and Seib (1993) for native potato starch containing 0.063% phosphorus, identified as the clearest starch. They reported that phosphates in corn and wheat starches, prepared with STPP at pH 6, exhibited a light transmittance over four times greater than that of raw potato starch. However, the transmittance for these starches was about 10% lower than that of native potato starch, which contradicts our findings. This discrepancy may be attributed to differences in starch source, phosphate distribution, and experimental conditions.

The optical transparency of potato starch pastes is governed by two fundamental structural determinants: (1) the stereospecific positioning of phosphate ester groups within the amylopectin framework, which dictates intermolecular electrostatic repulsion and hydration dynamics, and (2) the macromolecular architecture of amylose, encompassing its polymerization degree, chain-length distribution, and branching pattern. These molecular-scale features synergistically dictate the hierarchical organization of starch constituents during gelatinization, ultimately determining the wavelength-dependent light scattering properties of the colloidal system (Hu *et al.*, 2025). In contrast, crosslinking reduces paste transparency by reinforcing hydrogen bonding and restricting granule swelling, which leads to larger residual particles that scatter light more effectively (Mali *et al.*, 2001). This effect becomes more pronounced at higher pH levels, where accelerated crosslinking alters the granule structure and further reduces clarity (Kaur *et al.*, 2006).

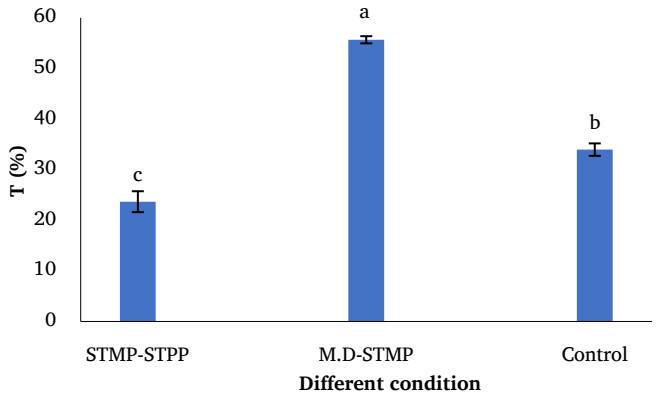

Fig. 2. Comparison of transparency of dual phosphorylated starches. Different letters indicate a significant statistical difference ($P < 0.05$). STMP: Sodium orthophosphate, sodium tripolyphosphate, STPP: Sodium trimetaphosphate, M.D.: Mono/Di phosphate Ratio, and Control: native potato starch.

Swelling power

According to Fig. (3), the highest swelling power was associated with the starch phosphorylated with a mixture of STPP and STMP. O'Brien *et al.* (2009) reported that phosphorylation under varying conditions did not result in significant changes in the swelling power of corn starch. Still, potato starch phosphorylated at pH 9 showed significantly higher swelling power than at pH 11. Saleh *et al.* (2020) reported that the swelling factor was linearly related to the concentration of the crosslinking agent used. The reduction in swelling factor is attributed to the formation of intermolecular bridges facilitated by phosphorus following the crosslinking reaction. Therefore, the decrease in swelling factor is related to forming intermolecular bridges by phosphorus after the crosslinking reaction. Heo *et al.* (2017) also reported that potato starch's swelling factor decreased as the crosslinking agent's concentration (STMP/STPP) increased from 0.0125 to 5%. However, no difference was observed between the 5 and 10% concentrations of the crosslinking agent. This may be attributed to the hardening of starch granules due to new covalent bonds acting as bridges between starch molecules.

Therefore, an increase in the concentration of phosphate compounds can lead to an increased water retention capacity due to the repulsion between negatively charged phosphate groups on starch molecules, which reduces intermolecular bonding forces. STMP, on the other hand, reduces the

distance between molecules by creating cross-links, minimizing water absorption (Mali *et al.*, 2001). Ultimately, in dual-phosphorylated starch, both crosslinking and repulsion between negatively charged phosphate groups occur. Thus, it appears that the effect of phosphate groups on the binding factors predominates, enhancing the swelling power of E1413 compared to the control starch.

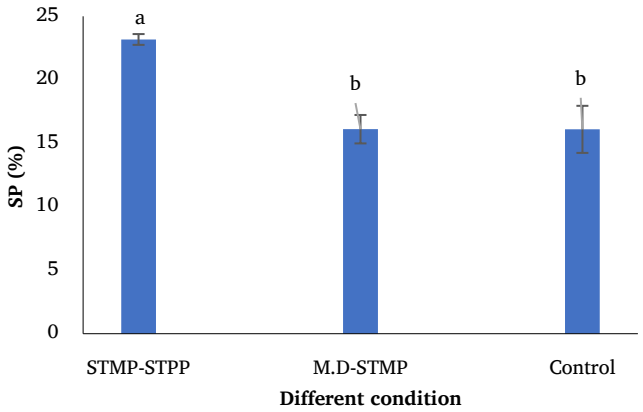

Fig. 3. Comparison of swelling power (%) of dual phosphorylated starches. Different letters indicate a significant statistical difference ($P<0.05$). STMP: Sodium orthophosphate, sodium tripolyphosphate, STPP: Sodium trimetaphosphate, M.D.: Mono/Di phosphate Ratio, and Control: native potato starch.

Thermal properties of optimized dual phosphorylated starch pastes

Heating a starch suspension in excess water gradually increases its viscosity due to gelatinization and molecular interactions. One of the methods for investigating changes in starch viscosity during thermal processing is using an RVA device. The basis of this equipment is heating along with applying shear force to a certain amount of the sample and recording the temperature and apparent viscosity over a specific time. This device applies the thermal process step by step (Bello-Pérez *et al.*, 2010). This section explores starch pasting characteristics and rheological changes during heating, aiming to predict the functional behavior of modified starch samples under food processing conditions.

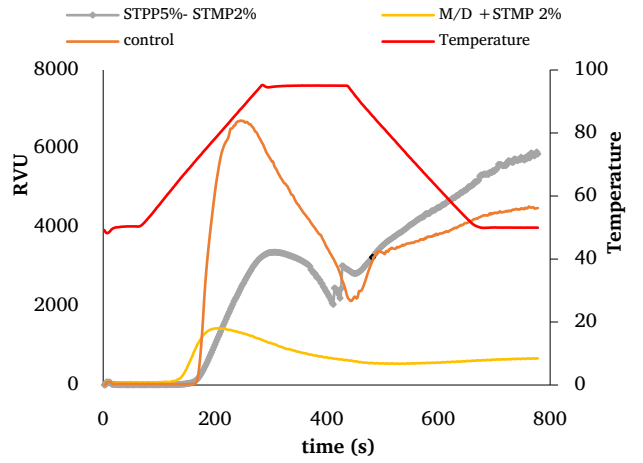

Fig. 4. Comparison chart of paste viscosity of dual-modified starches E1413. STMP: Sodium orthophosphate, sodium tripolyphosphate, STPP: Sodium trimetaphosphate, M.D.: Mono/Di phosphate Ratio, and Control: native potato starch.

Table 4. Comparison of viscosity indices of dual modified starch paste and order of starch chemical modification

Samples	Peak viscosity (cp)	Hold viscosity (cp)	Final viscosity (cp)	Break-down (cp)	Set-back (cp)
STPP5%-STMP2%	3373	2203	5866	1170	3663
M/D - STMP 2%	1443	536	677	907	141
Control	6717	2135	4496	4582	2361

STMP: Sodium orthophosphate, sodium tripolyphosphate, STPP: Sodium trimetaphosphate, M.D.: Mono/Di phosphate Ratio, and Control: native potato starch.

According to Fig. (4) and Table (4), the onset temperature and time for viscosity development in the M/D-STMP phosphorylated sample slightly decreased, which may be attributed to enhanced water absorption and disruption of crystalline regions, factors known to influence pasting behavior.

The temperature of reaching peak viscosity in phosphated distarch phosphate with STMP and STPP agent has increased compared to the control sample, which indicates greater thermal stability in this type of modified starch. In this section, it is clear that the modification of all three types of E1413 starches resulted in a decrease in peak viscosity, indicating the effect of crosslinking in preventing swelling and maintaining the granule in a better condition against heat and shear. This behavior indicates the effect of crosslinking in preventing swelling and maintaining the structural integrity of the granule under heat and shear, reflecting the improved resilience of the optimized dual phosphorylated starch pastes.

The peak viscosity of starch depends on various factors such as the degree of starch crystallinity, phosphorus content, granule integrity, starch solubility, the presence of salts and the presence of food additives (da Silva *et al.*, 2020; Nelles *et al.*, 2003; Ral *et al.*, 2008). The creation of crosslinks leads to the formation of bonds between the starch chains and increases the resistance of the starch granule to swelling. Crosslinking strengthens the granule structure and prevents water absorption by limiting the mobility of the starch chains in amorphous regions (Lin & Czuchajowska, 1998). It is also clear that the time to reach peak viscosity in the starch modified by STMP-STPP has increased. This indicates an increase in the stability of stirring and mechanical forces in the starch modified by STMP-STPP compared to the use of mono/diphosphate.

The effect of phosphate groups is due to the repulsion between the negatively charged substituent groups on the starch molecules, which reduces the intermolecular bonding and decreases the gelatinization temperature. The gelatinization temperature is also a function of the degree of crosslinking within the starch. However, it has been found that a low degree of crosslinking reduces the gelatinization temperature, and a higher degree of crosslinking increases it. Acetyl and phosphate groups reduce the gelatinization temperature by destabilizing the crystalline area of the starch granules (Ai & Jane, 2015).

The higher the viscosity of the starch suspension at a lower temperature, the greater the applicability of this type of sample in products with lower processing temperatures. The lower the pasting temperature, the lower the resistance

to the breakdown of the starch intramolecular hydrogen bridges. Cross-linked starches have higher plasticization temperatures due to the additional bonds in the chains, and as a result, E1413 modified starch is more resistant to granule rupture than unmodified starch (Nara & Komiya, 1983).

Based on the viscosity graphs of the M/D-STMP sample, the molecular integrity of the starch was preserved to a greater extent than in the control sample and the sample created with STMP-STPP. The presence of low viscosity poses challenges for using this sample (M/D-STMP) as a stabilizer (thickener); however, it permits its application as a texture improver and a water activity reducer in situations where high viscosity affects the mouthfeel of the product (Heidel, 1988).

The breakdown index is an important factor for investigating the resistance behavior of starch at high temperature and shear force simultaneously. An increased breakdown index indicates that the starch granules show reduced structural stability under mechanical agitation and elevated temperature, leading to a more pronounced viscosity drop during pasting (Nara & Komiya, 1983). Based on the viscosity graphs and Table 4, it is clear that the breakdown index in the dual modified E1413 has decreased, leading to an increase in stability in this group of samples. It has been previously reported that the viscosity breakdown can be reduced by crosslinking. Therefore, this observation can also infer crosslinking (Whistler *et al.*, 2012). Liu *et al.* (1999) showed that the resistance to shear force increases by creating crosslinks.

The final viscosity indicates the ultimate use of the starch and also the degree of retrogradation. Accordingly, it can be stated that the dual modified starch by STMP-STPP produces a higher final viscosity, which is more suitable from the point of view of thickening the product. By converting the hydroxyl groups of the starch molecules into larger ester or ether groups, more stable pastes and gels are formed, i.e. pastes and gels with a lower tendency to retrogradation (Rouault, 2017).

A starch that shows higher setback values has a higher tendency to retrogradation (Wang *et al.*, 2015). Retrogradation refers to the re-alignment and recrystallization of gelatinized starch chains, primarily amylose, upon cooling, which leads to the formation of more ordered structures and impacts the texture and shelf stability of starch-based products. During this process, amylose and amylopectin interact with the swollen starch granules in an organized manner, resulting in increased viscosity and gel formation. According to the comparative results presented in Table (4), the lowest tendency for retrogradation appears to be associated with mono- and di-phosphated starches, whereas the highest tendency is observed in starches modified using STMP-STPP. These trends are based on single measurements and should be interpreted accordingly.

The STMP-STPP samples also showed lower transparency than the other samples (Fig. 3). The transparency of starch pastes is closely related to starch retrogradation (Wang *et al.*, 2015). The decrease in transparency of STMP-STPP modified starch pastes is attributed to crosslinking reactions that reinforce intermolecular bonds and limit granule swelling. This results in a more compact granular structure with reduced light transmission, thereby lowering paste clarity (Eliasson, 2004).

Yogurt syneresis

Considering the physicochemical and thermal properties of E1413 starches (higher swelling power, higher final viscosity, and better thickening power) as well as ease of production, the modified starch STMP-STPP (at pH = 9.5) was selected for examination in stirred yogurt.

Syneresis reflects the instability of the gel matrix formed during yogurt production, typically resulting from rearrangement or compression of protein and polysaccharide networks, leading to water expulsion. This phenomenon adversely affects the visual and textural quality of the product, influencing consumer acceptance (Lobato-Calleros *et al.*, 2014). Starches-particularly modified ones-help reduce syneresis by enhancing the water-binding capacity of the gel and strengthening its internal structure, thereby stabilizing the weak gel network.

Based on the results obtained, which can be seen in Fig. (5), the modified starch E1413 (at 2%) had the lowest syneresis. This starch was statistically different compared to the yogurt sample with unmodified starch and syneresis was reduced. Therefore, E1413 starch can be useful in preventing syneresis.

The primary causes of syneresis in fermented products include the application of high temperatures during incubation, low solids content, or storage at unsuitable temperatures (Lucey, 2004). Amaya-Llano *et al.* (2008) used acid-treated starch in yogurt and showed that this type of starch did not have a significant effect on increasing the stability to syneresis. These researchers attributed the syneresis in the samples to the nature of the behavior of modified starches and reported the ability of starch to form stiff gels as a factor in controlling this undesirable indicator. They usually tend to rearrange and release water, which increases the amount of syneresis (Amaya-Llano *et al.*, 2008). Lobato-Calleros *et al.* (2014) used modified starch and native starch in stirred yogurt. These researchers demonstrated that both modified and native starch could reduce syneresis and enhance the hardness of yogurt texture.

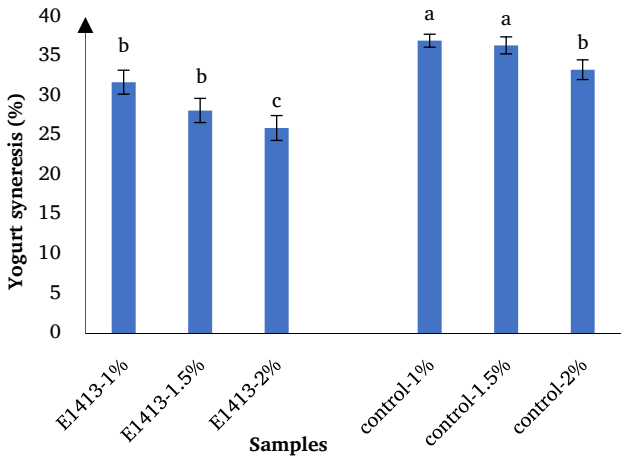

Fig. 5. Syneresis of yogurt samples produced by using dual phosphorylated starches (E1413) and controls at different concentration. Different letters indicate a significant statistical difference ($P < 0.05$).

Viscosity

Viscosity is one of the main parameters in fluid and semi-solid products. Smoothness of texture is a desirable sensory property in food emulsions such as dairy products. Smoothness can be related to creaminess and consistency, which depends on viscosity (Amaya-Llano *et al.*, 2008). Starches are used in yogurt production due to their ability to increase consistency, form gels, and retain water.

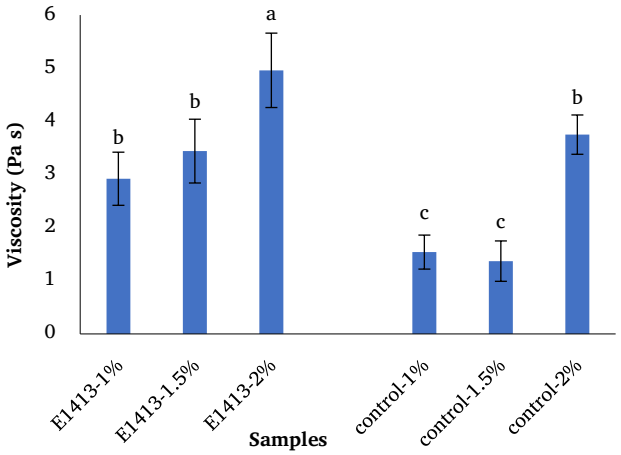

Fig. 6. Viscosity of yogurt samples produced by using dual phosphorylated starches (E1413) and controls at different concentration. Different letters indicate a significant statistical difference ($P < 0.05$).

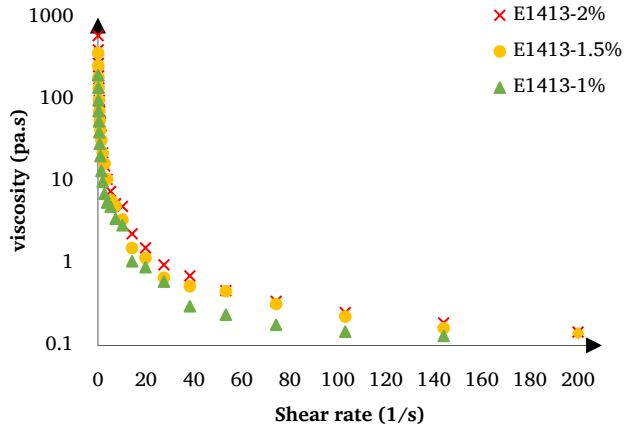

Fig. 7. Flow behavior of yogurt samples containing different amounts of E1413

By examining the viscosity of samples containing modified starch (Figs. 6 and 7), it was found that with increasing starch in the formulation, the viscosity increases. By comparing the viscosity of the modified starch samples, it is clear that the E1413 modified starch had the highest viscosity at a shear rate of 10/s at the 2% level. Phosphorylation introduces phosphate ester groups into the starch polymer chains, which increases their hydrophobicity and electrostatic repulsion, thereby enhancing the starch's swelling behavior and water-holding capacity. These changes increase the viscosity of yogurt. Studies have also shown that using acetylated starches in yogurt creates a stronger gel structure that increases the stability of the product during storage. This type of starch also reduces degradation under mechanical stresses and gives yogurt a more desirable consistency (Subroto *et al.*, 2023). Modified starches significantly increase the viscosity of yogurt. Starch's type and degree of modification affect its viscosity behavior (Schmidt *et al.*, 2001; Wong *et al.*, 2020). Abbas *et al.* (2017) used acetylated starch modified starch in low-fat yogurt. They found that the addition of acetylated starch led to an increase in viscosity and reduced syneresis.

According the Fig. (7) The rheological results reveal a clear concentration-dependent effect of E1413 on yogurt viscosity and shear-thinning behavior. Among the formulations, the 2% E1413 sample exhibited the most pronounced shear-thinning characteristics, indicative of a structurally robust yogurt matrix. The 1.5% sample displayed moderate viscosity (370 Pa.s) and intermediate thinning, while the 1% formulation had the lowest viscosity (198 Pa.s) and the highest degree of pseudoplasticity, marked by a 95.8% reduction in viscosity across increasing shear rates. This progressive decline in viscosity with decreasing starch content reflects diminished water-binding capacity and weaker starch-casein interactions. Importantly, all formulations retained measurable viscosity (>0.1 Pa.s) even under elevated shear stress (>20 Pa), confirming their non-Newtonian fluid behavior. These findings highlight the critical role of E1413 concentration in tuning yogurt rheology, offering formulation flexibility to achieve targeted mouthfeel, product stability, and processing performance.
Also, in this study, the viscosity of the samples was examined based on the Carreau-Yasuda model (Quintana Martinez *et al.*, 2022).

$$\eta = \eta_{\infty} + \frac{\eta_0 - \eta_{\infty}}{[1+(\gamma\lambda)^{\alpha}]^{n-\frac{1}{\alpha}}} \quad (6)$$

Where η is the viscosity (Pa.s), γ is the shear rate (s^{-1}), η0 is the viscosity at low shear rate (Pa.s) where this quantity is equal to 0.1, η∞ is the viscosity (Pa.s) at high shear rate, λ is the time constant (s), α is the model constant, and n is the flow index (dimensionless). In shear-thinning fluids ($n < 1.0$) (Quintana Martinez *et al.*, 2022), the flow behavior index (n) reflects the extent to which viscosity decreases as shear rate increases. A lower value of n indicates stronger thinning behavior, which is advantageous in processes that require reduced resistance during flow (Lobato-Calleros *et al.*, 2014).

The Carreau-Yasuda model graphs and tables are given in Table (5) and Fig. (8). As it is clear, all samples have shear thinning behavior and with increasing starch concentration, the thinning behavior decreases because the flow coefficient n increases and in other words, the resistance to shear increases. Increasing the concentration of starch enhances the overall viscosity of the yogurt system through densification of its polymeric network. Additionally, the elevated presence of starch molecules near milk casein promotes intermolecular interactions, which stabilize the gel structure and improve resistance to shear deformation. Also, the highest flow coefficient n is related to the modified starch E1413 at a 2% level. This result shows the greater resistance of this sample to environmental stresses and in other words, this sample has greater textural stability.

Yield stress is the applied stress at which the first irreversible deformation of the sample occurs. The study of yield stress is crucial for assessing the performance and processability of the product, as it can help predict the item's stability and long-term shelf life. According to the Carreau-Yasuda model, it is evident that the yield stress rises with

increasing starch concentration, which correlates with the enhanced textural stability of the sample. A higher yield stress prevents phase separation or degradation of the sample during transport. Consequently, the highest yield stress in the samples signifies greater structural stability of the gel in this instance (Bravo-Núñez *et al.*, 2019). Saleh *et al.* (2020) stated that differences in starch type can lead to differences in yield stress in the sample. Differences among the samples are attributed to variations in starch swelling capacity and water absorption. Furthermore, the release of amylose and the granular breakdown of starch, caused by heat or processing, affect the internal structure of the gel and lead to a reduction in yield stress. Structural integrity of the starch matrix, which can lower the yield stress by weakening internal cohesion.

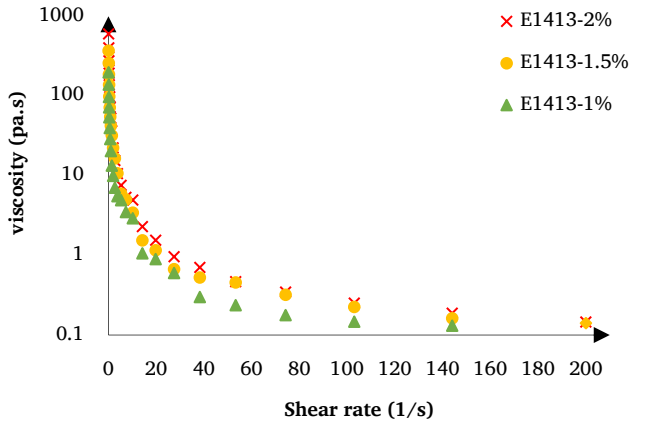

Fig. 8. Flow behavior of yogurt samples containing different amounts of E1413.

Table 5. Viscosity indices of yogurt samples containing different amounts of E1413

Sample	n (-)	α (-)	λ (s)	η_0 (Pa·s)	R^2
E1413-1%	0.36	5.62	61.89	1210	0.99
E1413-1.5%	0.47	4.49	95.19	7330	0.99
E1413-2%	0.66	2.97	335.8	11480	0.97

Texture hardness

The texture hardness of yogurt was investigated based on the penetration test. According to Fig. (9), results showed that increasing the starch content raised the force needed to penetrate the yogurt texture. Therefore, the highest hardness of the samples was noted with 2% E1413. The rise in hardness with higher starch concentration suggests that greater starch content offers more bonding points for water-fat interaction, which enhances the overall stiffness of the yogurt structure (Cui *et al.*, 2014). This observation is consistent with the findings of various studies that emphasize the role of starch as a gelling agent, helping to improve the texture and mouthfeel of dairy products (Saleh *et al.*, 2020). Studies have shown that modified starches can make yogurt texture creamier and smoother. This is achieved by forming a more elastic gel matrix that helps maintain the structure of yogurt during storage and transportation (Wong *et al.*, 2020).

In addition, the highest hardness of the samples was associated with E1413 at 2%. This was consistent with the viscosity test results. Starches phosphate, by adding phosphate groups, gain the ability to swell and absorb more water. These properties increase viscosity and improve the stability of the product containing this type of starch (Punia Bangar *et al.*, 2024). Cross-linked starches demonstrate enhanced resistance to heat and acid by forming cross-links between starch chains. These characteristics enable cross-linked starches to perform more effectively in products like yogurt that experience thermal and fermentation processes (Majzoobi *et al.*, 2014). Sandoval-Castilla *et al.* (2004) showed that using 1% modified corn starch in low-fat yogurt increased the hardness parameter of yogurt compared to the control sample. Abbas *et al.* (2017) reported that the addition of 2% acetylated starch adipate significantly increased the firmness of yogurt, even surpassing that of full-fat yogurt. In contrast, lower concentrations (1.5 and 1%) did not produce a statistically significant improvement in firmness. However, despite the enhanced texture at the 2% level, the taste was found to be undesirable by consumers.

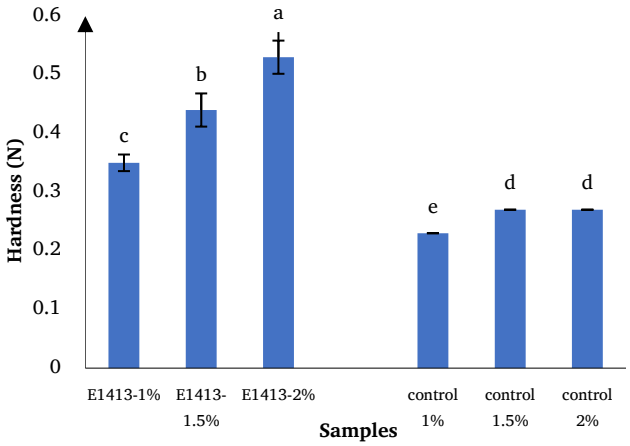

Fig. 9. Firmness of yogurt samples containing E1413 and native starches at different 1-2% concentrations. Different letters indicate a significant statistical difference ($P<0.05$).

Conclusion

The study successfully optimized the production conditions for phosphated distarch phosphate (E1413), revealing that phosphate compound concentration and mono/disodium phosphate ratio critically affected the degree of phosphorylation and physicochemical properties. Dual modification through phosphorylation and crosslinking enhanced starch granule stability against heat, acid, and shear, and improved paste viscosity and swelling power. The starch modified with STMP-STPP showed a longer time to reach peak viscosity, indicating greater stability against mechanical forces than mono/diphosphate-modified starch. This dual modification also resulted in higher final viscosity, making it more effective as a thickening agent. In contrast, the M/D-STMP sample maintained better molecular integrity but exhibited lower viscosity, limiting its use as a stabilizer while making it suitable for improving texture and reducing water activity. Theoretically, the work elucidates relationships between phosphorylation degree, crosslinking, and starch functionality. At the same time, practically, it offers a tailored approach to produce E1413 by using STPP-STMP that effectively reduces syneresis and improves viscosity and hardness in stirred yogurt, enhancing shelf life and texture. Recommendations include further investigation of sensory impacts, long-term storage stability, and

production scalability, as well as exploring applications in diverse food matrices to expand the utility of these modified starches.

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Author contributions

Hamed Saberian: Writing the draft of the manuscript, Presenting the research idea and study design, Revising and editing the manuscript, Supervising the study, Approval of the final version, Funding acquisition and Project administration; **Ali Forouhar**: Data collection, Data analysis, Data analysis and interpretation, Presenting the research idea and study design.

Conflicts of interest

There is no conflict of interest based on the writers.

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