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A new way to expand the application of starch and tung oil: tung oil anhydride modified starch

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Abstract

In order to expand application of starch and tung oil, tung oil anhydride modified potato starch (TOA starch) was prepared in this work. The structural property, degree of substitution, pasting properties, rheological properties and emulsifying ability of the TOA starch were investigated. Fourier-transform infrared spectroscopy analysis proved formation of modified starch, and degree of substitution of TOA starch was 0.021 ± 0.001. Compared with natural potato starch and its gel, TOA starch had lower pasting temperature and breakdown while higher trough viscosity, final viscosity and setback, and its gel had higher recovery capability. In addition, oil-in-water emulsion was prepared using TOA starch. Its particle size and ζ-potential was 551 ± 9.7 nm and -44 ± 0.1 mV, respectively. More importantly, it had similar storage stability with emulsion stabilized by octenyl succinic anhydride modified starch, which was stable stored for 20 d. These result further confirmed feasibility of modifying starch using tung oil anhydride and expanded application of starch and tung oil.

Keywords: starch; tung oil anhydride; chemical modification; physicochemical properties; emulsion.

Practical Application: Modified starch with high emulsifying capacity.

1 Introduction

Starch is a natural polymer that composed of glucose linked by α-1, 4 and α-1, 6, and is the most abundant storage polysaccharide in plants (Hu & Miao, 2020). In addition, starch is the main component of most foods as well as a raw material in industrial production. Starch and its products have been widely used in many industries, such as food, paper, textile, plastic, cosmetics, adhesives and pharmaceutical industries (Cao et al., 2022; Deng et al., 2022; Khantarate et al., 2022; Qin et al., 2022). However, native starch cannot always withstand extreme processing conditions, such as high temperature and repeated shear. That limited the use of starch in many industries. Therefore, starch was widely modified to enhance or inhibit its inherent properties or to endue its specific properties to meet the requirements of industrial applications. In particular, chemical modification of starch was considered to be very promising, because it usually had greater impact on starch compared with other modification methods, such as physical modification (Masina et al., 2017; Punia, 2020; Zhu, 2017).

Tung oil is a kind of vegetable oil that is extracted from the fruit of tung tree, which is widely planted in China. Tung oil and its products have been widely used in the field of material science, medicine and feed, etc. (Banaszkiewicz & Czechowski, 2020; Ito et al., 1983; Lee et al., 2002; Liu et al., 2019b; Tang et al., 2019). However, the development and utilization of tung oil and its products were still insufficient. A large number of tung tree fruits and tung oil were wasted every year. Therefore, it is urgent to expand the application of tung oil and its products. In recent years, it was found that tung oil anhydride which was formed by the reaction of tung oil and maleic anhydride could be used to modify other substances, including starch (Bao et al., 2004; Xiong et al., 2013). For example, Xiong et al. (2013) changed the mechanical properties and physical behaviors of polylactide and starch blends using tung oil anhydride as a plasticizer. Based on this, it was speculated that modification of starch using tung oil anhydride is a possible direction to expand the application of starch and tung oil.

However, structural, physicochemical and function properties of the tung oil anhydride modified starch have not been systematically characterized. In this work, tung oil anhydride modified potato starch was prepared, and its fourier-transform infrared spectroscopy, degree of substitution, pasting properties, rheological properties and emulsifying ability were investigated.

2 Materials and methods

2.1 Materials

Potato starch was purchased from Meelunie BV (Netherlands, total starch content: 97%, protein content: < 1%, fat content: < 1%). Octenyl succinic anhydride (OSA) modified starch (OSA starch, PURITY GUM 2000) was donated by Ingredion China Limited (Songjiang, Shanghai, China) and its degree of substitution was 0.031. Tung oil anhydride (TOA) was purchased from Weng Jiang Reagent (China, 99% purity). Other reagents used were of analytical grade and above.

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2.2 Preparation of tung oil anhydride modified starch

About 30.0 g potato starch was dissolved in 100 mL water. After adjusting the pH to 8.5 with NaOH solution (3.0%, w/w), the TOA (1.5%, water basis) was slowly dropped into the starch solution within 2 h. During reaction, the pH of the starch solution was maintained at 8.2-8.5 using the above NaOH solution. The reaction was allowed to proceed for 3 h at 40 °C. After reaction, the pH of the starch solution was adjusted to 6.5 with diluted HCl solution (0.1 mol/L). The TOA modified starch was then precipitated by adding 1800 mL isopropanol (90%, w/v) and washed several times with distilled water and isopropanol. Finally, the precipitate was oven-dried at 40 °C for 24 h, ground and passed through a 100 mesh nylon sieve to obtain the TOA modified starch. The TOA modified potato starch was named TOA starch.

2.3 Fourier-transform infrared spectroscopy analysis

The TOA starch and natural potato starch were spectra scanned by attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) (Nicolet 5700, Thermo Electron Inc., USA) (Wang et al., 2020). Specifically, starch was placed on the ATR plate and tightly pressed. The spectrum was acquired in the wavelength range 4000-500 cm^{-1} with the resolution ratio 4 cm^{-1} and was an overlap of 32 scans.

2.4 Determination of the degree of substitution

Degree of substitution (DS) of the TOA starch was measured using the titration method (Plate et al., 2012). Specifically, about 5.0 g TOA starch was dispersed by agitation for 30 min in 25 mL HCl-isopropyl alcohol solution (2.5 mol/L). Isopropyl alcohol solution (90%, v/v) (100 mL) was then added with agitation for another 10 min. The suspension was filtered and the residue was washed with 90% isopropyl alcohol solution until no chloride ions were detected by 0.1 mol/L $AgNO_3$ solution. The residue was oven-dried at 40 °C for 24 h, and then passed through a 100 mesh nylon sieve.

The TOA starch (1.0 g) was dispersed in 75 mL deionized water with stirring until the TOA starch was completely dissolved. The solution was titrated with 0.1 mol/L standard NaOH solution using phenolphthalein as an indicator. Natural potato starch was titrated as a control. The DS was calculated using Equation 1:

$$DS = \frac{0.162 \times \left(C \times V\right)/W}{1 - \left[0.390 \times \left(C \times V\right)/W\right]} \tag{1}$$

Where C and V was the concentration (mol/L) and volume (mL) of NaOH solution used, respectively; W (g) was the weight of the TOA starch.

2.5 Pasting properties analysis

Pasting profiles of the TOA starch and natural potato starch were determined by Rapid Visco Analyzer (RVA-TecMaster, Newport Scientific Pt. Ltd., Australia) based our pervious method (Hu et al., 2018). Specifically, starch (3.0 g, 12% moisture basis) was added to the aluminum test canister of Rapid Visco Analyzer, followed by adding 25 mL of deionized water, procedural stirring, heating (50-95 °C) and cooling. A plot of paste viscosity versus time was used to determine peak viscosity, trough viscosity, final viscosity, breakdown value and setback value.

2.6 Rheological properties analysis

Rheological properties of the TOA starch and natural potato starch were characterized according to our previous method (Guo et al., 2020b). Specifically, starch suspension (3.0%, w/v) was heated at 95 °C for 30 min with stirring to obtain the completely gelatinized starch gel. Dynamic shear rheometer (Kinexus, Malvern Instruments, Ltd., UK) was then used to characterize rheological properties (2.6.1-2.6.2) of the starch gel. This dynamic shear rheometer had a cup-and-bob geometry and Peltier temperature control unit. In this study, diameter of the bob and the cup was 25 and 27.5 mm, respectively. The test temperature was set as 25 °C, and the starch gel was relaxed at 25 °C for 5 min in the geometry before test. A solvent trap was covered on the geometry to limit moisture evaporation of the starch gel.

Assay of frequency sweep

Storage moduli (G′) and loss moduli (G″) of the starch gel were measured under frequency from 0.1-10 Hz. The strain was constant at 1% (within the linear viscoelastic region).

Assay of in-shear structural recovery

In-shear structural recovery of the starch gel was calculated by a three-step shear flow test. Specifically, shear rate of the first stage, the second stage and the third stage was constant at 1 s^{-1} for 120 s, 300 s^{-1} for 60 s and 1 s^{-1} for 180 s, respectively. Shear recovery value was calculated by ratio between the average apparent viscosity value (η) of the first 120 s of the third stage and that of the first stage.

2.7 Preparation of oil-in-water emulsions using succinic anhydride modified starch and tung oil anhydride modified starch

The coarse oil-in-water emulsion was prepared by blending 10.0 g of oil phase (corn oil) and 90.0 g of aqueous phase (OSA starch or TOA starch solution, 1% w/v) using a laboratory disperser homogenizer (ShangHai Angni Instruments & Meters Co., Ltd., China) for 5 min at a speed of 15000 rpm. The coarse oil-in-water emulsion was then recirculated three-times through a high-pressure microfluidizer (NCJJ, Langfang General Machinery Manufacturing Co., Ltd., Langfang, China) at an operational pressure of 80 MPa to obtain the final oil-in-water emulsion. The OSA starch stabilized emulsion was named OSA emulsion, the TOA starch stabilized emulsion was named TOA emulsion.

2.8 Emulsifying ability analysis

Observation of micromorphology

Micromorphology of the TOA emulsion and OSA emulsion was observed using an optical microscope. Specifically, a 100 μL emulsion was placed on glass slide, covered with cover glass, and observed under a 40× objective.

Assay of particle size and ζ-potential

Particle size and electrical charge (ζ-potential) of the TOA emulsion and OSA emulsion were determined using a particle electrophoresis instrument (Zetasizer NanoZSP, Malvern Instruments, Worcestershire, UK). Specifically, the emulsion was diluted using buffer solutions that had same pH value as the emulsion, and detected in the instrument.

Assay of storage stability

The TOA emulsion and OSA emulsion were stored at 25 °C. The 0.02% (w/v) sodium azide was added into the emulsions to prevent the growth of microorganisms.

2.9 Statistical analysis

Results were expressed as mean ± standard deviations of triplicate analyses for each sample. The statistical analyses were performed using SPSS (version 22.0, SPSS Inc., USA). A comparison of the means was ascertained by Tukey's test at 5% level of significance using one-way analysis of variance (ANOVA).

3 Results and discussion

3.1 Fourier-transform infrared spectroscopy

The FTIR was widely used to analyze structure of starch due to its high sensitivity to molecular structure (Dankar et al., 2018). Figure 1 was the FTIR spectra of the TOA starch and natural potato starch. They showed the same characteristic peaks at 3400 cm^{-1}, 2900 cm^{-1} and 1645 cm^{-1}. Peaks at 2900 cm^{-1} and 3400 cm^{-1} were due to CH_2 deformation and OH bonds, respectively (Kačuráková & Mathlouthi, 1996). Peak at 1645 cm^{-1} was assigned to water molecules absorbed in the amorphous region and the stretching vibration of the C=O band (amide I) (Kizil et al., 2002). In addition, the TOA starch showed diffraction peaks in 1570 cm^{-1} and 1723 cm^{-1} (Figure 1), which was different from the natural potato starch. It was reported that peak of 1723 cm^{-1} was ascribed to the carbonyl C=O stretching vibration of an ester group, and peak of 1570 cm^{-1} was an symmetric stretching of vibration of a carboxyl group (Wang et al., 2020). Therefore, the result confirmed that the potato starch was successfully modified by the TOA.

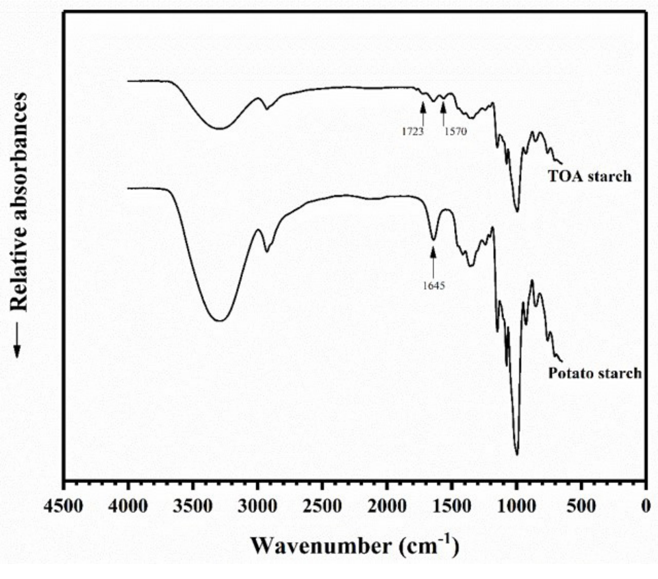

Figure 1. Fourier transform infrared spectroscopy spectra of the TOA starch and natural potato starch.

3.2 Degree of substitution

The DS value is an important index, which could reflect the chemical modification degree of starch. It was reported that the higher the DS value, the greater the degree of starch modification (Wang et al., 2020). Therefore, the DS of the TOA starch was determined. The DS of the TOA starch was 0.021 ± 0.001. This result was in accordance with the FTIR analysis, which also confirmed the formation of the TOA starch.

3.3 Pasting properties

It was widely known that altered structure of starch always had effects on functional properties of starch, such as pasting properties (Hu et al., 2018). The above results confirmed that the structure of potato starch was changed by the TOA. Therefore, it was investigated whether pasting properties of the potato starch were changed by the TOA. Plots of paste viscosity of the TOA starch and natural potato starch versus time were shown in Figure 2. Their peak viscosity, trough viscosity, final viscosity, breakdown value and setback value were shown in Table 1. Compared with the natural potato starch, the TOA starch had lower pasting temperature and breakdown value ($p < 0.05$), while had higher trough viscosity, final viscosity and setback value ($p < 0.05$).

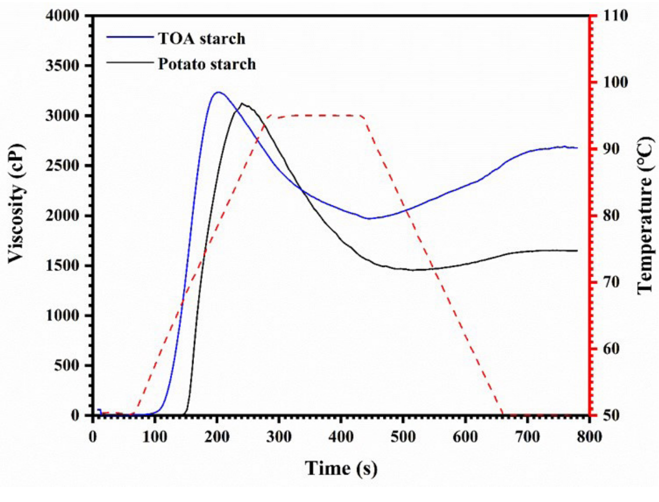

Figure 2. Pasting properties plots of the TOA starch and natural potato starch.

Table 1. Pasting properties of the potato starch and TOA starch.

Sample	Pasting temperature (°C)	Peak viscosity (cP)	Trough viscosity (cP)	Breakdown (cP)	Final viscosity (cP)	Setback (cP)
Potato starch	67.80 ± 0.07^{a}	3172 ± 67^{a}	1457 ± 3^{b}	1715 ± 64^{a}	1634 ± 21^{b}	177 ± 23^{b}
TOA starch	58.80 ± 0.57^{b}	3234 ± 1^{a}	1981 ± 18^{a}	1253 ± 18^{b}	2675 ± 5^{a}	694 ± 23^{a}

All data were expressed as mean ± standard deviations (n = 3). Values in the same column followed by different superscript letters were significantly different at $p < 0.05$ by Tukey's test.

Pasting temperature of starch reflects the minimum temperature needed for starch gelatinization. Decreasing pasting temperature suggested that the TOA starch was easier to gelatinize than the natural potato starch. It was reported that the pasting temperature of starch was related to its difficult degree of absorbing water at low temperature and damage degree of starch granules (Asmeda et al., 2016; Bello-Flores et al., 2014; Quintero-Castaño et al., 2020; Remya et al., 2018). Therefore, it was speculated that the TOA starch granules might be damaged in the modification process, which caused the TOA starch easier to absorb water at low temperature. Damage on the surface of starch granule caused by the esterification modification was also reported in other researches (Bello-Flores et al., 2014; Quintero-Castaño et al., 2020; Remya et al., 2018; Sharma et al., 2016), which supported our speculation.

Breakdown value reflects the thermal stability of starch to some extent. Decreasing breakdown value suggested that the TOA starch had higher thermal stability than the natural potato starch, which could withstand greater heating and shearing. It was reported that the breakdown value of starch was related to its dispersion and difficult degree of particle breaking (Asmeda et al., 2016; Hu et al., 2018; Liu et al., 2019c). Therefore, there were two possible reasons in decreasing breakdown value of the TOA starch. First, the TOA increased dispersion of the starch (evidenced by its lower pasting temperature and peak time). Second, the TOA that attached to the outside of starch granules might make the starch granules more difficult to break, though the starch granules might be damaged by the modification as stated above. Sandhu et al. (2015) also observed that the breakdown value of potato starch was decreased after esterification. However, the breakdown value of other varieties of starch tended to increase after esterification (Punia et al., 2019; Wen et al., 2020), thus the result might be a unique phenomenon of potato starch.

Setback value reflects the difficult degree of short-term retrogradation of starch to some extent. Increasing setback value suggested that the TOA starch was more prone to short-term retrogradation than the natural potato starch. It was reported that the setback value of starch was related to rearrangement of amylose which was leached out from the swollen starch granules during cooling (Asmeda et al., 2016). Thus, it was inferred that the increase of the setback value was due to the damage of the TOA starch granules during modification process as stated above, which promoted the leach of amylose from swollen starch granules during cooling.

In a word, the TOA modification had great influences on pasting properties of starch. Low pasting temperature and breakdown value of the TOA starch indicated that the TOA starch could saving heating energy and had the potential of application at the condition of high temperature.

3.4 Rheological properties

Frequency sweep

Rheological properties of starch are also a very important index to evaluate application of starch (Guo et al., 2020b). Frequency sweep is a non-destructive method for characterizing response of starch gel to impact (high frequency) and gradual loading (low frequency) under small strain condition (within the linear viscoelastic region). Plots of the G’ and G” of the TOA starch gel and natural potato starch gel versus frequency were shown in Figure 3a. In the result, the G’ of both the starch gels was much higher than their G” in the test range. The G’ reflects the elasticity of starch gel and the G” reflects the viscosity of starch gel (Zhang et al., 2020). The result indicated that both the starch gels were predominantly elastic. In addition, the G’ and G” of both the starch gels slightly increased with the increasing of frequency, indicating that both the starch gels were weak gels, and the interior of both starch gels was covalently jointed (Liu et al., 2019a). Finally, there was no significantly difference in the G’ and G” between the TOA starch gel and natural potato starch gel, indicating that the TOA did not change the viscoelasticity of potato starch. This result was consistent with the pasting properties result (the TOA starch and natural potato starch had same peak viscosity).

In-shear structural recovery

In general, starch gel is typically thixotropic. Therefore, recovery capability of starch gel under reversible thixotropic was worth studying. In-shear structural recovery curves of the TOA starch gel and natural potato starch gel were shown in Figure 3b. Viscosities of both the starch gels decreased obviously under high shear rate (300 s^{-1}), which indicated that structure of the starch gels was disturbed. After returning to low shear rate (1 s^{-1}), viscosities of the TOA starch gel and natural potato starch gel recovered to 66% and 31%, respectively. This result suggested that the TOA starch gel had higher recovery capability than the natural potato starch gel and could withstand greater shearing. The result was consistent with the pasting properties result (the TOA starch had greater thermal stability than the natural potato starch). In addition, it was found in our previous studies that increase in the recovery capability was related to damage of starch granule to some extent (Guo et al., 2020b; Liu et al., 2019a). Therefore, this result was in accordance with the pasting properties result, which further confirmed that a certain degree of starch damage was occurred in the modification process. In sum, high recovery capability of the TOA starch gel indicated

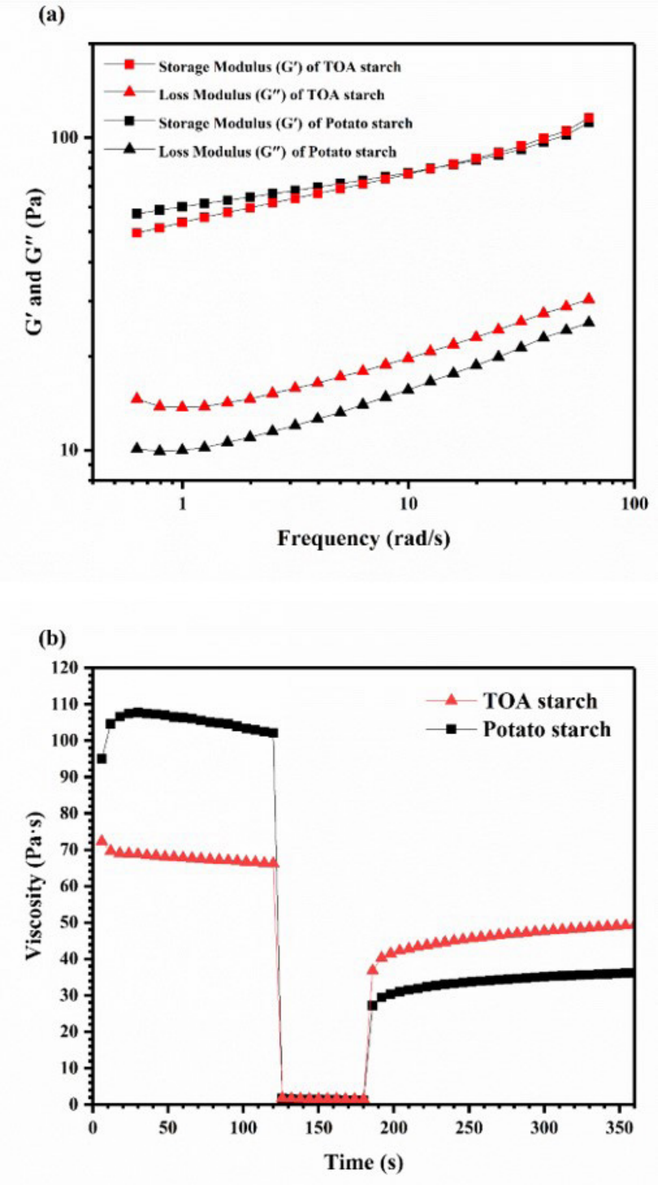

Figure 3. Frequency sweep (a) and in-shear structural recovery (b) plots of the TOA starch gel and natural potato starch gel.

that the TOA starch gel had the potential of application at the condition of repeated shear.

3.5 Emulsifying ability

It was widely known that the starches modified by the anhydrides usually had excellent emulsifying ability and were often used as emulsifiers (Lin et al., 2018; McClements, 2011; McClements & Jafari, 2018; Yan et al., 2019b; Yan et al., 2019c). Based on the above point of view, it was speculated that the TOA starch also has good emulsifying ability. Therefore, emulsifying ability of the TOA starch was measured and compared with that of the OSA starch in this work. The OSA starch was used as control because it was currently recognized anhydride modified starch with the best emulsifying ability.

Micromorphology

Firstly, micromorphology of the TOA emulsion and OSA emulsion was observed to evaluate their emulsifying ability. Both the TOA emulsion and OSA emulsion showed homogeneous status, and no obvious oil droplet accumulation was observed (Figure 4). The result indicated that both the TOA emulsion and OSA emulsion had good stability. In other words, both the TOA starch and OSA starch had good emulsifying ability. In addition, the TOA emulsion showed bigger droplets than the OSA emulsion. It was reported that emulsion with small particle size had relatively high stability (Guo et al., 2020a; Ma et al., 2020; Zhu et al., 2018). Therefore, the result indicated that stability of the TOA emulsion was weaker than that of the OSA emulsion to some extent. In other words, emulsifying ability of the TOA starch was weaker than that of the OSA starch to some extent.

Furthermore, it was well known that the higher the DS value of the esterified starch, the stronger the emulsifying ability (Xie et al., 2020). As mentioned above, the DS value of the TOA starch and OSA starch was 0.021 and 0.031, respectively. Therefore, compared with the OSA starch, the lower DS value of the TOA starch might be responsible for its weak emulsifying ability. Unfortunately, the TOA starch with the same DS value as the OSA starch was not obtained in this study. Therefore, their emulsifying ability at the same DS value was still worthy of further comparison.

Particle size and ζ-potential

Particle size and ζ-potential are very important indexes to evaluate the quality of emulsion (Yan et al., 2019a; Yan et al., 2019b; Yan et al., 2019c). Therefore, particle size and ζ-potential of the TOA emulsion and OSA emulsion were measured and shown in Figure 5. Z-average diameter of the TOA emulsion and OSA emulsion was 551 ± 9.7 nm and 273 ± 0.5 nm, respectively. Their z-average diameters reached nanometer level (< 600 nm). Generally, the emulsion with nanometer size had good stability (McClements, 2011). Therefore, it was inferred that the TOA emulsion and OSA emulsion had good stability, which was consistent with the micromorphology result. In other words, both the TOA starch and OSA starch had good emulsifying ability. In addition, the OTA emulsion showed bigger z-average diameter than the OSA emulsion. Consistent with the micromorphology result, this result also indicated that stability of the TOA emulsion was weaker than that of the OSA emulsion. In other words, emulsifying ability of the TOA starch was weaker than that of the OSA starch.

The ζ-potential of the TOA emulsion and OSA emulsion was -44 ± 0.1 mV and -34 ± 1.6 mV, respectively (Figure 5). Generally, a high charge of emulsion indicated that electrostatic repulsion was involved in the stabilization of emulsion (Chung et al., 2018; Dai et al., 2019). Thus, the result suggested that electrostatic repulsion was one of the force of the TOA starch and OSA starch to stabilize emulsion. In addition, it was reported that steric hindrance was one of the force acting on starch stabilized emulsion (Guo et al., 2020a; Yan et al., 2019b; Yan et al., 2019c). Therefore, it was inferred that steric hindrance was also one of the force for the TOA starch and OSA starch stabilized

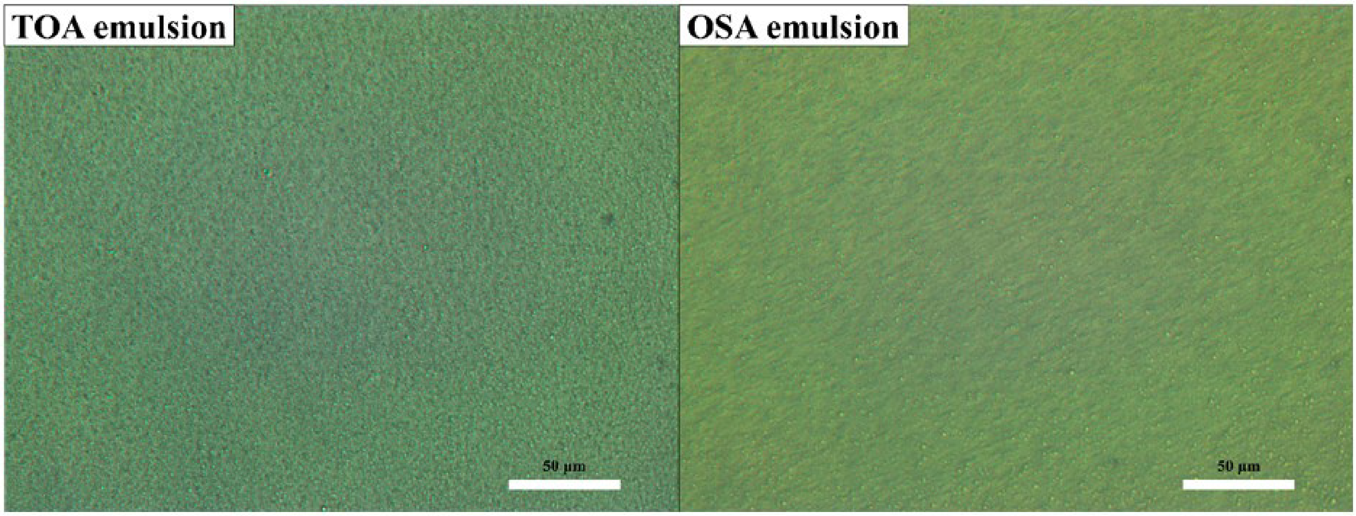

Figure 4. Micromorphology of the TOA emulsion and OSA emulsion.

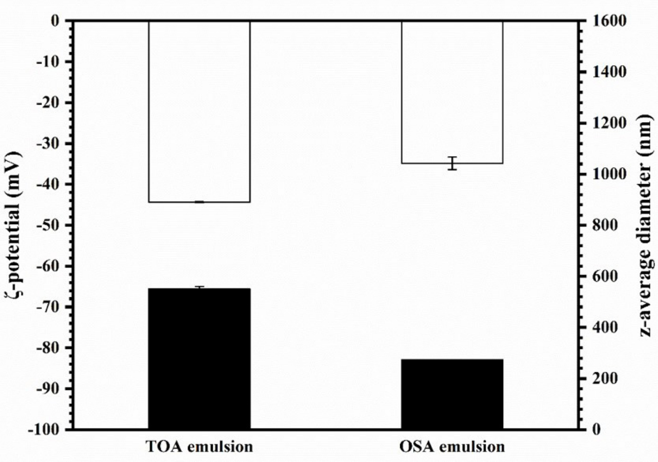

Figure 5. Particle size and ζ-potential of the TOA emulsion and OSA emulsion.

emulsion. All in all, it was inferred that the TOA starch was same with the OSA starch, which stabilized emulsion manly through electrostatic repulsion and steric hindrance. It was also reported that electrostatic repulsion and steric hindrance were two main force of the emulsion stabilized by the anhydrides modified starch (Wang et al., 2020; Xie et al., 2020; Yan et al., 2019c), which supported our speculation.

Storage stability

Storage stability is one of the most important indicators to evaluate the quality of emulsion (Gómez-Luría et al., 2019). Therefore, storage stability of the TOA emulsion and OSA emulsion was investigated and shown in Figure 6. Both the TOA emulsion and OSA emulsion showed excellent storage stability. Both the TOA emulsion and OSA emulsion could be storage for 20 d, though the TOA emulsion was not as good as the OSA emulsion in other indicators (such as micromorphology and particle size). Generally, most application needs could be meet if emulsion could be stored for 15 d or more (Gómez-Luría et al., 2019; Guo et al., 2020a; Pan et al., 2019), because emulsions used in vernacular food products were commonly consumed within 2-7 d. Therefore, the result proved that the TOA emulsion had enough stability to be applied in practice.

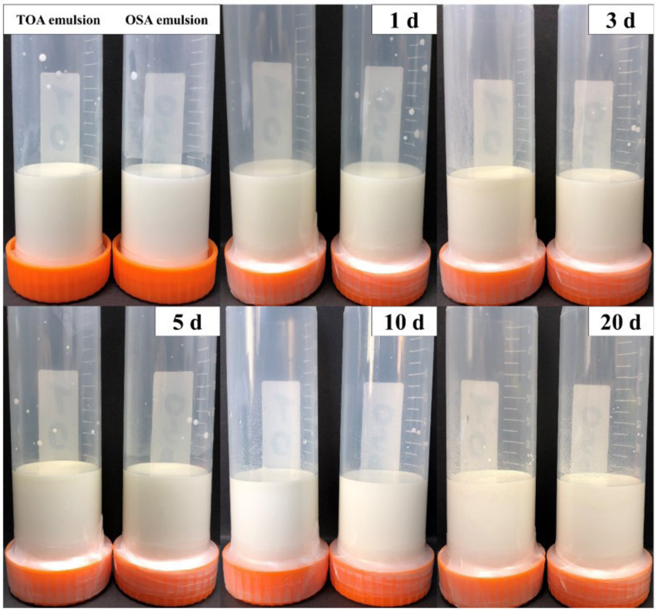

Figure 6. Storage stability of the TOA emulsion and OSA emulsion.

4 Conclusion

Tung oil anhydride modified potato starch (TOA starch) was prepared in this work. Fourier-transform infrared spectroscopy analysis proved the formation of modified starch, and degree of substitution of the TOA starch was 0.021 ± 0.001. Compared with the natural potato starch and its gel, the TOA starch had lower pasting temperature and breakdown value while higher trough viscosity, final viscosity and setback value, and its gel had higher recovery capability. For used as emulsifier, the TOA starch stabilized emulsion could be storage for 20 d, which was same with the emulsion stabilized by octenyl succinic anhydride modified starch. These result suggested that the TOA starch could saving heating energy and had the potential of application at the conditions of high temperature and repeated shear. More importantly, the results indicated that emulsifying ability of the TOA starch was enough to meet the needs of practice. This study further confirmed feasibility of modifying starch using tung oil anhydride and expanded application of starch and tung oil. However, toxicology of the TOA starch was still worthy of further study if the TOA starch needs to be used in food industry.

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