INTRODUCTION

Over the past few decades, with the large-scale integration of closed animal husbandry operations and fruit cultivation, a state-of-the-art waste management approach is needed to make rural development economically viable and at the same time environmentally friendly. In addition to potential environmental threats, traditional waste management systems such as landfill and incineration can lead to energy consumption and waste of land resources. Thus, there are expanded interests in the conversion from livestock waste and rural waste to renewable energy and chemicals. Taking biofuel and platform chemical production technologies to the farm provides an opportunity for the agricultural sector to reduce their reliance on imported fossil fuels while improving the soil, water, and air quality.

Fruit peel, especially citrus peel, is a common agricultural waste that can be easily collected in rural areas. Moreover,
the citrus peel can be used as an ingredient for animal feed, bio-oil, charcoal, essential oil, adsorbent, and integrated process for the production of ethanol, biogas, and limonene. In recent years, the utilization of citrus peel has become a hot research area due to its easy availability. However, in China, most citrus peel is disposed. According to Beatriz et al., the citrus peel contains 16.9% soluble sugar, 3.75% starch, 9.21% cellulose, 10.5% hemicellulose, and 42.5% pectin, and it can be converted into pentose and hexose via hydrolysis. Collectively, citrus peel is a potential and promising resource to produce levulinic acid (LA) and furfural (FF). LA and FF are the two important platform chemicals targeted in this study. Global LA and FF market demand is estimated to reach 3820 ton by 2020 and 1434 million USD by 2022, respectively. In contrast with this huge market demand, the prices of LA and FF are relatively high. It is considered an effective way to reduce the production cost by using agriculture waste to prepare platform chemicals that are easy to recover. FF is the raw material of up to 1600 chemical products and is regarded as a promising alternative to the petrochemical industry. It derives from the dehydration of pentose, and therefore, any lignocellulosic biomass which contains arabinose and xylose can serve as a raw material for FF production. LA is the simplest γ-oxocarboxylic acid. It possesses a ketone carbonyl group and an acidic carboxyl group, which makes it easy for LA to react with various functional groups to form different derivatives. The most important synthetic route for the production of LA is an acid-catalyzed multistep conversion of polysaccharide content of biomass. It includes hydrolysis of polysaccharides to monosaccharides, dehydration of hexose to HMF, and rehydration of HMF to produce LA.

The production of FF and LA from biomass hydrolysis has been reported. Girisit et al. obtained a 19.4 wt% yield of LA by hydrolysis of bagasse, which equates to the production of 194 kg of LA from 1 dry tonne of sugar cane bagasse. Amudhavalli et al. studied the hydrolysis reaction of four cellulose-rich biomass materials to produce LA; under optimal reaction conditions (423 K, 1 mol/L HCl, 2 hours) the yields of LA obtained from Cicer arietinum, cotton, Pinus radiata, and sugarcane bagasse were 32.6, 44.0, 19.0, and 36.5 wt%. However, the yield of FF during these conversion processes is maintained at a low level, and the production of LA and FF from pectin has never been reported before. The FF yield can be effectively increased by adding organic solution to its original solution; DMSO is a good reaction solvent for dehydration of hexose and pentose. It is favorable for the formation of FF and LA precursor HMF. Kimura et al. found out that through situ 13C NMR and DMSO, α-fructose was converted first into 3,4-dihydroxy-2,5-di(3-hydroxymethyl)tetrahydrofuran; there is no double bond in the ring, subsequently into 4-hydroxy-5-hydroxymethyl-4,5-dihydrofuran-2-carbaldehyde having one double bond through dehydration, and finally into 5-hydroxymethyl-2-furaldehyde (5-HMF) having two double bonds.

In this study, we investigated the hydrolysis behavior of citrus peel, the main products such as LA and FF were obtained under mild conditions. Compared with the typical lignocellulosic biomass such as straw, the soluble oligosaccharides in citrus peel make it have a higher platform chemical yield under milder conditions. In addition, world citrus production has exceeded 100 million tons, of which 35% is used for subsequent processing. Citrus peel is the by-product of citrus processing, accounting for about 25%-40% of the citrus weight. Therefore, the comprehensive utilization of citrus peel will improve the economic benefit of citrus processing plant and reduce pollution.

2 | MATERIALS AND METHODS

2.1 | Source of materials

The citrus peel used in this experiment was purchased from a citrus deep-processing enterprise located in Fujian Province. The citrus peel used in this experiment came from the most common citrus species in southern China. LA, glucose, FF, and sulfuric acid were purchased from Aladdin. Cellulose pectin was purchased from Shanghai Xianding Biotechnology Co., Ltd. The hydrochloric acid, acetone, calcium chloride, acetic acid, 10% silver nitrate solution, ethanol, trypsin, and potassium ferrocyanide required for the determination of the chemical composition of the peel were purchased from Shanghai Macklin Technology Co., Ltd. (MACKLIN).

2.2 | Citrus peel hydrolysis process and chemical composition analysis method

The citrus peel was fully dried in an oven at 80°C and then ground. After passing through a 40-m sieve, it is ready for use. 100 mg of citrus peel and 5 mL of 0.15 mol/L sulfuric acid solution were added to a glass pressure reactor (15 mL) that was heated up to a predetermined temperature (120-200°C) in a CEM Discover SP single-mode microwave synthesis system. The speed of magnetic stirring was kept at 500 rpm. When the reaction time reached the pre-set time, the reactor was removed from the heating equipment and cooled down by forced air convection. The solution obtained from the reactor was filtered through a 0.22-μm filter and diluted 10-fold with distilled water. It was then analyzed by high-performance liquid chromatography (HPLC). Further, the ash, sucrose, cellulose, pectin, and hemicellulose contents in citrus peel were determined with reference to the Chinese national food ingredient testing standards.
2.2.1 | Determination of LA and FF yield

The diluted hydrolysate was filtered and analyzed on an UltiMate 3000 HPLC system (Thermo Scientific, USA). The retention times of LA and FF were 15.265 and 43.75 minutes, respectively. The concentration of FF and LA in the diluted hydrolysate, which was determined by comparison with the standard calibration curve, was recorded as \( c_1 \) and \( c_2 \), respectively. The mass yield of FF and LA were calculated as follows:

\[
Y_1 (L) = 100\% \times \frac{c_1 \times 10 \times 5}{m_1} \\
Y_2 (F) = 100\% \times \frac{c_2 \times 10 \times 5}{m_1}
\]

where the mass yield of LA is \( Y_1 (L) \) and the mass yield of FF is \( Y_2 (F) \).

2.2.2 | Measurement of citrus peel components

Take a crucible of appropriate size and place it in a muffle furnace, then burn it at 550°C for 0.5 hour. After cooling to below 200°C, take it out from the muffle furnace and cool it to room temperature in a desiccator. Weigh, then burn, and reweigh until reaching a constant weight, where the mass was recorded as \( m_1 \). Add 2.00 g of citrus peel powder to the crucible, which was placed on an electric furnace with asbestos mesh. Heat the crucible on a small fire until the sample was fully carbonized. Then, put it in a muffle furnace and burn it at 550°C for 4 hours. After cooling to below 200°C, take it out and put it in a desiccator for 30 minutes. If there are carbon particles before weighing, add a small amount of water to the sample and burn it to complete ash, then weigh. Repeat the calcination to constant weight, where the mass was recorded as \( m_2 \). The calculation formula of the result is as follows:

\[
X_1 (A) = 100\% \times \frac{m_2 - m_1}{2.00}
\]

where \( X_1 (A) \) is the ash content of citrus peel.

5.00 g of samples were put in a 150-mL Erlenmeyer flask; 100 mL of water and 1% trypsin were added. Then, the samples were hydrolyzed at 37°C for 2 hours. Heat the hydrolysates at 95°C for 1 hour then transfer it to a 250-mL volumetric flask. Allow it to stand for 30 minutes after constant volume. The filtrate was analyzed by HPLC for sucrose content (\( X_2 (S) \)).

2.00 g of citrus peel powder was put in an oven at 80°C. The sample was placed in a 250-mL iodine volumetric flask, and 150 mL of 2 mol/L HCl was added. Then, the samples were incubated at 105°C for 48 minutes. After being taken out, the products were filtered with a glass funnel until the filtrate was neutral. The residue was washed with distilled water after filtration the next day until the filtrate had a pH of 6.5-7.0. The filtered residue was transferred into a crucible (\( m_3 \)) with constant weight and dried in an oven at 80°C to a constant weight (\( m_5 \)). The calculation formula is shown in the following equation, the hemicellulose content is \( X_3 (H) \), and the cellulose content is \( X_4 (C) \).

\[
X_3 (H) = 100\% \times \frac{m_1 - m_3 + m_2}{2.00} \\
X_4 (C) = 100\% \times \frac{m_3 + m_4 - m_2 - m_5}{2.00}
\]

15.00 g of citrus peel was used; 150 mL of water was added for the first time to heat up to 100°C for extraction for 1 hour. 75 mL of water was added for the second time to heat up to 100°C for extraction for 0.5 hour. The two extracts were then combined. The total volume of the extract was recorded as \( V \). The extract was filtered until the solution was clear. 30 mL of the extraction solution was placed in a 500-mL beaker, 100 mL of 0.1 mol/L sodium hydroxide solution was added, and the mixture was stirred for 0.5 hour. Then, 50 mL of 1 mol/L acetic acid solution was added, and 25 mL of 1 mol/L calcium chloride solution was slowly added while stirring. After 1 hour of reaction, the reaction mixture was heated and boiled for 5 minutes. The reaction mixture was filtered while it was still hot. The mass of the funnel was recorded as \( m_1 \). The reaction mixture was washed with hot water until no chloride ions (tested with a 10% silver nitrate solution). The filter residue together with the funnel was dried in a 105°C oven to a constant weight, and the total mass of the filter residue and the funnel was recorded as \( m_2 \). The calculation formula is as follows. The coefficient for converting pectinate to pectin is 0.9233, and the pectin content is \( X_5 (P) \).

\[
X_5 (P) = 100\% \times 0.9233 \times \frac{(m_2 - m_1) \times V}{30 \times 15}
\]

3 | RESULTS AND DISCUSSION

3.1 | Citrus peel composition

Table 1 shows the distribution of the main components of citrus peel. The results suggested that the citrus peel is rich in sucrose (9.62%) and pectin (23.1%). Sucrose
is an important precursor of LA. Under acidic conditions, sucrose is first hydrolyzed to produce six-carbon sugar (C6) and then isomerized and dehydrated to form 5-HMF. 5-HMF is further hydrated to form LA and formic acid.16,17 The basic structure of pectin is a long chain of α-galacturonic acids linked by α-1,4 glycoside bonds, which usually exist in methylated form. Figure 1 shows the typical structure of pectin, and it also contains some polysaccharides composed of six-carbon sugars (such as rhamnogalacturonan, galactan), which are also good precursors for preparing LA.18 Compared to wood and straw-based biomass, the cellulose and hemicellulose content of citrus peel is at a lower level, and the cellulose and hemicellulose contents are 18.84% and 17.01%, respectively. The lower hemicellulose content leads to a lower yield of FF in the final hydrolysate.19 Other hydrolysates include essential oils, flavonoids, limonoid, and lignin. The results of citrus peel component distribution are similar to what has been previously reported.20 Based on this result, we propose that the citrus peel has good prospects of preparing LA.

### 3.2 Effect of reaction temperature

Firstly, the effect of different temperature conditions on the mass yield of LA and FF in pure water was studied. In the sulfuric acid hydrolysis system, the cellulose and sucrose in citrus peel were firstly hydrolyzed to hexose, which was further dehydrated to HMF, then rapidly converted to LA and formic acid.21 Meanwhile, the pectosan in citrus peel is hydrolyzed to pentose, which is further dehydrated to produce FF. As shown in Figure 2, it can be clearly found that the temperature greatly affects the yield of LA and FF. When the temperature was set to 170°C, the yield of LA increased to 17.9% at 45 minutes, and gradually increased with the increase in temperature. After 60 minutes, the dominant reaction becomes a side reaction that consumed LA and causing its yield to decline.12,23 When the reaction time was 60 minutes and the temperature was 190°C, the highest yield of LA obtained was 21.9%. Under similar conditions, the yield of LA in wheat straw is 11%, and the yield of LA in potato peel is 15% in the research of Novodárszki et al.24 On the contrary to the case of LA, the yield of FF began to show a significant downward trend from 15 minutes and on. Danon et al25 proposed that when the temperature is above 160°C, the reactivity of converting monosaccharides to FF will be greatly improved, resulting in a large amount of FF formation in a short period of time from the beginning of the reaction. This made the FF rapidly participate in side reactions such as esterification and polycondensation after the FF is formed. In short, a suitable reaction temperature needs to be chosen for the hydrolysis of biomass to obtain the highest yield of LA and FF.

### 3.3 Effect of solvent composition

To study the effect of different solvents on the yield of FF and LA, further experimental conditions were performed on the hydrolysis of citrus peel under different solvent. Two different organic solvent systems, namely the water-butanolone system and the water-DMSO system, were selected for comparison with pure water. As can be seen from Figure 3, in the solvent system of water-DMSO, the yield of LA was significantly reduced, while the yield of FF was improved compared with that in pure water. The FF yield reached a maximum of 9.1% at 140°C. At 190°C, the maximum yield of LA was 8.8%, which was far lower than 21.9% of the pure water system. In the water-butanolone system, the change in LA and FF yield with temperature was similar to that in the water-DMSO system. The maximum yield of FF was 7.1% at 140°C, and the maximum yield of LA was 14.8% at 190°C. The comparison showed that the FF yield in the water-DMSO system was the highest among the three solvents. This is due to the inhibitory effect of water-DMSO on side reactions, which can effectively inhibit the side reactions leading to FF degradation in this solution system, such as the condensation reaction between FF and intermediates, the self-condensation reaction of FF, and the condensation reaction of FF and LA, which were the main reasons leading to the decrease of FF yield. Based

| Composition | Cellulose | Hemicellulose | Lignin | Pectin | Reducing sugar | Flavonoids | Ash |
|-------------|-----------|---------------|--------|--------|----------------|------------|-----|
| Content (wt.%) | 18.84 | 17.01 | 9.16 | 23.1 | 9.62 | 2.56 | 4.32 |
on the results of the three systems, it can be found that the relationship between the yield of LA and the temperature is similar to that of FF. In the range of 120-190°C, the yield of LA monotonically increases with temperature, reaching a maximum at 190°C. The highest yield of LA in the pure water system, water-DMSO system, and water-butanol system were 21.9%, 8.8%, and 7.1%, and the highest yields of FF were 5.4%, 8.8%, and 7.1%, respectively. Therefore, a suitable solvent system needs to be selected to achieve higher target product selectivity under mild conditions.

The effect of citrus peel concentration on LA and FF yields was further investigated, and the content of citrus peel was 100 mg, 200 mg, and 400 mg, respectively. The results are shown in Figure 4. When the amount of citrus peel was 100 mg, the yield of LA reached a maximum at 60 minutes and then began to decrease. When the amount of citrus peel was increased up to 200 mg and 400 mg, the yield of LA decreased. The yield of LA of citrus peel at three initial concentrations showed similar properties, so the reaction system was not suitable for the efficient conversion of citrus peel to LA at a high initial concentration. When the content of citrus peel was 100 mg, 200 mg, and 400 mg, the maximum yields of LA were 21.5%, 20.1%, and 15.9%, respectively. When the amount of citrus peel was 400 mg, the maximum yield of LA was significantly reduced, and a rapid downward trend occurred after 60 minutes. In this case, the concentrated substrate will produce a large amount of intermediate, so LA will undergo intense side reactions. Omari et al. and

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FIGURE 1  Typical structure of pectin

FIGURE 2  Curves of the yield of LA (A) and FF (B) vs time-based on dry-based biomass. Citrus peel 100 mg, H$_2$SO$_4$ concentration 0.15 mol/L, reaction time 15, 30, 45, 60, 90, 120 min
Tabasso et al.\textsuperscript{27} suggested that low acid concentration and low substrate concentration can effectively inhibit the formation of by-products.\textsuperscript{22,27} Therefore, to obtain a satisfactory yield of LA at a high substrate loading concentration, the reaction time and corresponding operating conditions need to be selected carefully.

### 3.4 Effect of citrus peel components

In order to investigate the reason for the high yield of LA in the aqueous product of citrus peel, we further studied the hydrolysis products of molding compounds of major components in citrus peel. Among the components of citrus peel, FF is mainly converted from hemicellulose and pectin, and LA is mainly converted from cellulose and sucrose. The results are shown in Figure 5. The overall yield of FF is at a relatively low level in pectin hydrolysate, and the highest yield is 26.05%, which is reached at 180°C at 30 minutes. With the increase in reaction time, the yield of FF showed similar properties to that of citrus peel. In the first 30 minutes, the yield of FF showed a monotonous increase. After 60 minutes, the side reactions were beginning to dominate and the yield of FF continued to decrease. The main components in pectin could be divided into four categories, galacturonic acid, rhamnogalacturonic acid, arabinogalactan, and xylose galacturonic acid.\textsuperscript{28} We speculated that galactose and rhamnose from the later stage of pectin hydrolysis were further converted to FF, but the contents of these two pentoses in pectin were relatively low, which resulted in a low FF yield.\textsuperscript{29} In addition, the FF yield from xylose can reach up to 66.32%; according to the component analysis of citrus peel, pectin and hemicellulose were the main components that can be converted into FF. By the contents of pectin and hemicellulose and their conversion into FF, it was estimated that half of the FF in peel hydrolyzed products came from the hydrolysis of hemicellulose, and nearly half of the FF produced from citrus peel came from pectin. It can be seen from the yield of LA in

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure3}
\caption{The yield of LA and FF under different temperature and solvent conditions. (A) Water:DMSO = 1:4, (B) pure water, (C) water:butanone = 1:4 (volume ratio), citrus peel 100 mg, H\textsubscript{2}SO\textsubscript{4} concentration 0.15 mol/L, reaction time 60 min. Effect of citrus peel concentration}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure4}
\caption{The yield of LA and FF with different initial content of citrus peel. Pure water system, H\textsubscript{2}SO\textsubscript{4} concentration 0.15 mol/L, reaction temperature: 190°C, solvent: 5 mL, reaction time: Reaction time 15, 30, 45, 60, 90, 120 min}
\end{figure}
the hydrolysis of sucrose and glucose that the LA produced by citrus peel mainly comes from cellulose and sucrose.

3.5 | Surface morphology of citrus peel residue

In the experiment of preparing LA and FF by hydrolysis of citrus peel, due to the occurrence of unavoidable side reactions, a large amount of depolymerization residue (humin) were generated. It is necessary to develop the use-value of humin to increase the practical feasibility of this transformation process. In order to better understand the structure and surface morphology of the depolymerized residue, we performed the SEM analysis on the depolymerized residue under two conditions. The results are shown in Figure 6. These two reaction conditions correspond to the case where the maximum yield of LA was obtained in the pure water system (A) and the case where the yield of FF was the highest (B). It can be seen from Figure 6 that the surface macrostructures of the two residues are significantly different. At 190°C, the original fiber structure of the citrus peel was hardly observed in the residue, and the residue distributed in the form of particles. Moreover, the particles exhibited an irregular porous structure. At 140°C, the residues showed a sheet-like fiber structure, and they were tightly connected. It can be found that the fiber structure in the citrus peel component is more likely to be destroyed and removed under severe reaction conditions, which can also explain the higher yield of LA obtained at higher reaction temperatures.

4 | CONCLUSION

In this study, we investigated the hydrolysis behavior of citrus peel for the production of LA and FF. In a pure water
system, the maximum yield of LA was 21.9% at 60 minutes and 190°C. In water-DMSO and water-butanone mixtures, the yield of LA decreased significantly, while the yield of FF was higher than the pure water system. Within the first 60 minutes of the three systems, the yield of LA showed a monotonously increasing trend. Afterward, the side reaction began to dominate and the yield of LA continued to decrease. Next, the pectin obtained from citrus peel pectin also has the potential to be converted into FF and LA. The highest yield of LA obtained from pectin was 13.1%. The surface morphology of the depolymerized residue indicated that severe reaction conditions could help destroy the original fiber structure of citrus peel.

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