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Research Article

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Study on by-products synthesis of powder coating polyester resin catalyzed by organotin

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Abstract: In the production of powder-coating polyester, a strong odor emanates from the water, prompting us to investigate its composition and origin. We studied the process of powder coating epoxy/polyester 50/50, which employs butyl tin tris (2-ethyl hexanoate) as a catalyst. The esterified water was extracted using ethyl acetate. It was analyzed via GC–MS to identify the side reactions and by-products that arise during the tin-catalyzed esterification process. Then we discussed their formation mechanism. Our results indicate that butyl tin tris (2-ethyl hexanoate) triggers side reactions such as cyclization and transesterification, leading to the production of by-products such as heterocyclic compounds (1,4-dioxane), anhydride compounds (propionic anhydride), and others. Notably, the unpleasant odor was found to be derived from propionic anhydride. These findings provide insights into the chemistry of tin-catalyzed esterification and highlight the importance of addressing the formation of unwanted by-products in the production of powder-coating polyester.

Keywords: butyl tin tris (2-ethyl hexanoate), side reactions, cyclization, by-products, powder-coating polyester

1 Introduction

In recent years, the issue of unpleasant odors emanating from powder-coating polyester (1,2) factories has become a major concern. The strong and irritating smell has a significant impact on the surrounding environment. Due to production process and equipment constraints, a large portion of the exhaust gases generated during the polyester production process are directly released into the air after simple spraying. However, the effectiveness of this method is limited and fails to adequately address the odor issue. Moreover, this approach does not meet emission standards (3) and results in air pollution, which poses a threat to human health and the environment. Although some enterprises have installed exhaust gas incineration systems, the high costs associated with such systems are prohibitive. Therefore, a more comprehensive understanding of this issue is required to facilitate targeted treatment and improve the situation.

There are many sources of odor. The process of the esterification reaction is as follows: During the esterification reaction of polyester for powder coating, some raw materials will be carried out by the esterification of the water, which can lead to the formation of by-products that contribute to the odor. The catalyst (4) used in the process can also form reaction intermediates, altering the reaction rate and causing side reactions like diol (acid) cyclization (5) under its action. Additionally, they are also carried out by the esterification of the water. Therefore, it is critical to study the by-products produced during polyester synthesis catalyzed by the catalyst to better understand the sources of odor and develop appropriate solutions.

There has been limited research on the by-products of polyester production. Sun et al. (6) investigated the removal of organic compounds and the microbial community in a two-stage alternating anaerobic/aerobic (A1/O1/A2/O2) process for treating wastewater from polyester resin production. The study found that the process was effective in removing the majority of esters, alkanes, phenols, acids, and alcohols from the wastewater. However, two hazardous and persistent compounds, 5,5-dimethyl-1,3-dioxane and 1,4-dioxane, were still present as major components in the final effluent. These compounds are by-products of the esterification process and are carried by water.

1,4-Dioxane (7) is an organic compound that is typically generated as a by-product during the production of polyesters and other polyoxy compounds. It is recognized as a probable carcinogen (8) and is both persistent and

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mobile. Polyester manufacturing involves two main stages: esterification and polycondensation, which use terephthalic acid and ethylene glycol as starting materials. When glycol is heated and distilled with dehydration catalysts like sulfuric acid, diethylene glycol – with the structure $HO–CH_2–CH_2–O–CH_2–CH_2–OH$ can be condensed, leading to the formation of 1,4-dioxane. The improper disposal of industrial waste streams or accidental solvent spills can easily contaminate water (9) sources with this hazardous compound.

1,4-Dioxane is known to be highly resistant to biotic and abiotic degradation (10) due to its heterocyclic structure, making it difficult to remove from contaminated sites. It is also highly soluble in water, which limits the effectiveness of attenuation mechanisms like volatilization and adsorption. However, research has demonstrated that 1,4-dioxane can be biodegraded as a sole source of carbon and energy (11), and economically efficient biological treatment options are available. For instance, Han et al. (12) investigated the potential of a laboratory-scale upflow biological aeration filter packed with tire chips for treating 1,4-dioxane.

The potential for other by-products emitting irritating odors during polyester esterification water production has not yet been analyzed. Butyl tin tris (2-ethylhexanoate) (13) is commonly used as a catalyst in polyester production and was utilized in this study for the synthesis of powder-coating polyester. The purpose of this experiment was to analyze the composition of the esterified water and to identify the types of by-products produced by the action of the tin compound catalyst under this formulation. We classified these by-products and inferred the reaction mechanisms of some feedstock side reactions under the tin-like compound catalyst formulation. The results of this study can serve as a reference for the industry to optimize the production process and reduce by-products while controlling pollution in a scientific manner.

2 Experiment

2.1 Raw material

Neopentyl glycol (industrial grade, Wanhua Chemical Group Co., Ltd), ethylene glycol (industrial grade, Shandong Hengxin Chemical Co., LTD), diethylene glycol (industrial grade, Guangzhou Canlian Chemical Co., Ltd), terephthalic acid (industrial grade, Jinan Aochen Chemical Co., Ltd), propylene glycol (industrial grade, Dow Chemical Co., Ltd), trihydroxymethyl propane (industrial grade, Nantong Baichuan New Material Co., LTD), butyl tin tris (2-ethylhexanoate) (industrial grade, Yunnan Tin TinChem Technology Co., Ltd), and ethyl acetate (AR, Tianjin Fuyu Fine Chemical Co.).

2.2 Analytical instrument and parameter setting

The analysis was performed using the Agilent Intuvo 9000 GC Gas chromatography–mass spectrometer, which is equipped with a DB-WAX chromatographic column (30 m × 0.25 mm × 0.25 µm) packed with polyethylene glycol. The instrument was set with the following gas chromatograph parameters: the inlet temperature was 250°C and the injection volume was 1 µL. The carrier gas used was helium gas with a purity of 99.999%. The initial temperature was set at 35°C and held for 5 min. It was then increased to 80°C at a rate of $5°C·min^{-1}$ and held for 3 min. Subsequently, the temperature was increased to 110°C at a rate of $5°C·min^{-1}$ and then to 250°C at a rate of $20°C·min^{-1}$. The total analysis time was 30 min.

The MS conditions were set as follows: electron ionization was used as the ionization source with an ionization energy of 70 eV. The transmission line and ion source were maintained at temperatures of 280°C and 230°C, respectively. The solvent delay time was set to 1 min. The MS2 scan mode was employed for ion selection.

2.3 Formula of resin

The composition of raw materials for epoxy polyester powder coating formula is subject to constant change due to various factors such as raw material source, price, and product performance requirements. There are obvious differences in the composition of the formulas of different companies. Some companies may use trimethyl propane instead of trimellitic anhydride and diethylene glycol in place of other diols.

The resin products used in the preparation of polyester/epoxy powder coating typically have a transparent solid appearance that is colorless or light yellow. They have a softening point within the range of 95–115°C and an acid value of 68–80 mg $KOH·g^{-1}$. These products exhibit a glass transition temperature (T_g) above 50°C, making them suitable for the preparation of a 50/50 (mass ratio) polyester/epoxy powder coating (14) (Table 1). If the amount of polyester used is greater than the amount of epoxy, the acid value of the polyester should fall below this range. Conversely, if the amount of polyester used is less than the amount of epoxy, the acid value of the polyester should be higher than this range. However, such polyester

Table 1: Epoxy/polyester 50/50 formula

Material	**Mass (g)**
Neopentyl glycol	112.77
Ethylene glycol	99.62
Diethylene glycol	93.98
1,2-Propanediol glycol	53.65
Trimethyl propane	4.70
Terephthalic acid	635.28
Butyl tin tris (2-ethyl hexanoate)	1.00

resins with high acid values are costly and not practical for use in epoxy–polyester powder coatings.

2.4 Methods for synthesizing epoxy/polyester 50/50

In this experiment, a mixture of 635.28 g (3.82 mol) of terephthalic acid, 4.70 g (0.035 mol) of trimethyl propane, 112.77 g (1.08 mol) of neopentyl glycol, 99.62 g (1.61 mol) of ethylene glycol, 93.98 g (0.89 mol) of diethylene glycol, 53.65 g (0.71 mol) of 1,2-propylene glycol, and 1 g of catalyst was added into a 1-L four-mouth flask. The mixture was stirred at 300 rpm and the condenser was turned on. The temperature was raised from room temperature to 250°C and the weight of the water produced was measured every 10 min. The reaction was stopped when the temperature reached 250°C and water samples were taken.

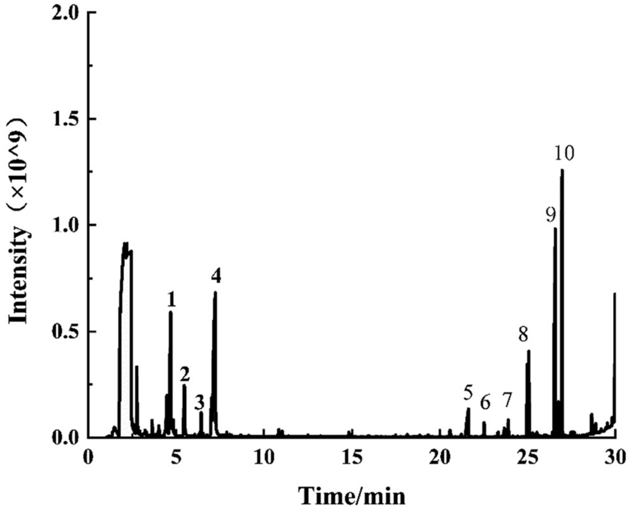


Figure 1: GC–MS determination of total ion flow diagram of esterified dehydrated water: (1) 1,4-dioxane; (2) 5,5-dimethyl-1,3-dioxane; (3) propionic anhydride; (4) 5,5-dimethyl-1,3-dioxane-2-ethanol; (5) 1,2-propanediol glycol; (6) propane-1,2-diol 1-butyrate; (7) propanoic acid, 2-methyl-, 2-hydroxyethyl ester; (8) isobutyric acid 2-ethyl-3-hydroxyhexyl ester; (9) neopentyl glycol; and (10) isobutyl 2-ethylhexanoate. Spectral type is electron impact. The area percentage of selected by-products was greater than 5.

2.5 Extraction of esterified water

We employed a pipette to transfer 10 mL of water sample and 10 mL of ethyl acetate to a 60 mL separation funnel. The mixture was vigorously shaken and allowed to settle into two distinct liquid layers. Subsequently, we collected the top layer of the ethyl acetate extract phase into an injection bottle for further analysis.

We chose ethyl acetate as our extraction agent and conducted gas chromatography–mass spectrometry (GC–MS) analysis using the heating-up procedure parameters depicted in Figure 1.

3 Results

3.1 Water sample

The water sample exhibits clarity and transparency along with a pungent odor under the catalytic action of butyl tin tris(2-ethylhexanoate).

3.2 Total particle flow diagram

There are numerous qualitative methods for analyzing organic matter, and one of the most convenient and efficient techniques is the GC–MS method. In this experiment, the GC–MS method is adopted for qualitative analysis of organic matter. After heating the sample according to Table 1, the distillate was extracted using ethyl acetate and analyzed using GC–MS. The results are presented in Figure 1, and the bar graphs for the by-products are shown in Figures 2–11.

4 Discussion

4.1 Catalytic mechanism of butyl tin tris (2-ethylhexanoate)

4.1.1 Step one

The reaction between dibasic acid and butyl tin tris (2-ethylhexanoate) involved the breaking of the O–H bond

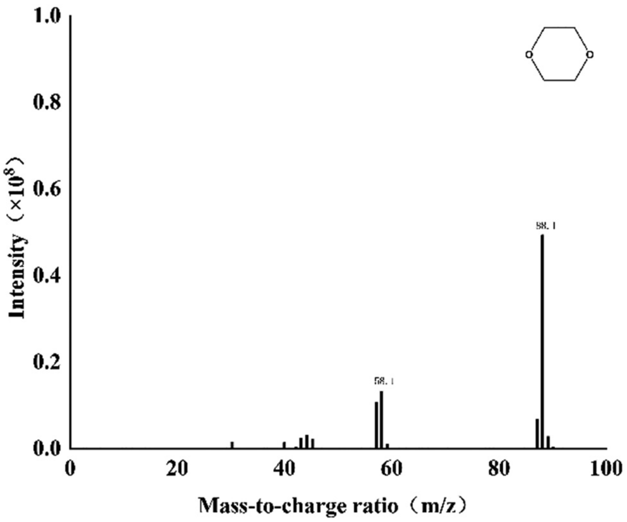


Figure 2: Bar graph of 1,4-dioxane. (1) 1,4-Dioxane (the mass spectral library matching score is 90.31), *m*/*z* 58.1 (intensity is 0.13×10^8), 88.1 (intensity is 0.49×10^8).

in the former and the Sn–OX bond in the latter. The resulting fragments then formed an O–Sn bond, yielding a molecule of 2-ethylcaproic acid (Figure 12).

4.1.2 Step two

The O–Sn bond and the O–X bond both broke, leading to the formation of an OH–Sn bond. This reaction resulted in the creation of a molecule of a tin compound (Figure 13).

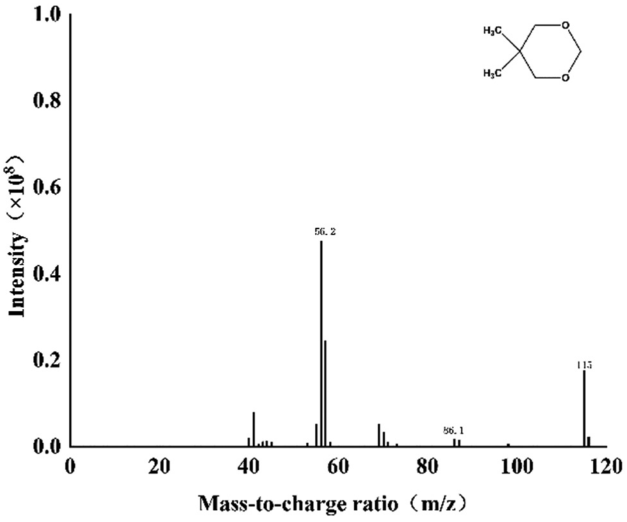


Figure 3: Bar graph of 5,5-dimethyl-1,3-dioxane. (2) 5,5-Dimethyl-1,3-dioxane (the mass spectral library matching score is 84.24), *m*/*z* 56.2 (intensity is 0.47×10^8), 86.1 (intensity is 0.02×10^8), 115 (intensity is 0.18×10^8).

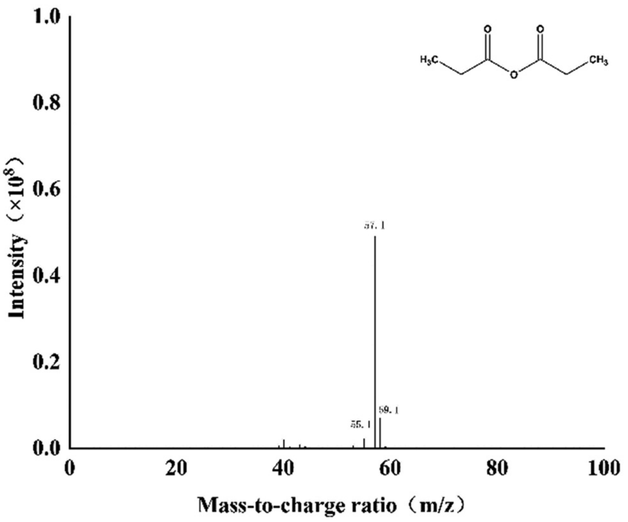


Figure 4: Bar graph of propionic anhydride. (3) Propionic anhydride (the mass spectral library matching score is 85.94), *m*/*z* 55.1 (intensity is 0.02×10^8), 57.1 (intensity is 0.49×10^8), 58.1 (intensity is 0.07×10^8).

4.1.3 Step three

The O–Sn bond and the O–H bond of the diol both broke, resulting in the formation of butyl tin tris (2-ethylhexanoate) and a molecule of H_2O (Figure 14).

4.2 By-products classification

In this experiment, the by-products were classified into four main groups: heterocyclic compounds, esters, acids,

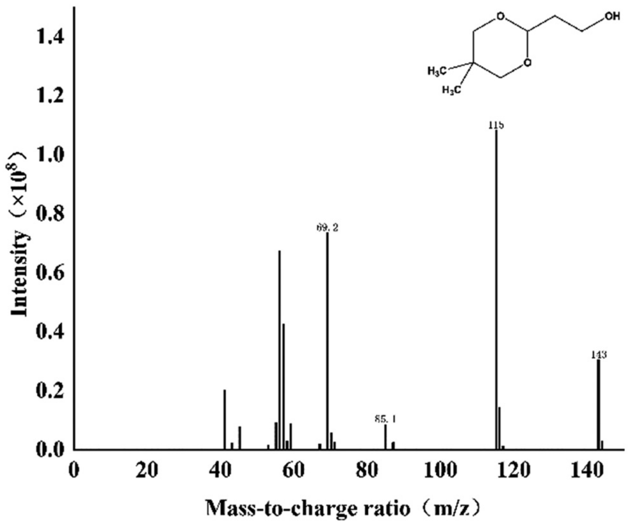


Figure 5: Bar graph of 5,5-dimethyl-1,3-dioxane-2-ethanol. (4) 5,5-Dimethyl-1,3-dioxane-2-ethanol (the mass spectral library matching score is 86.91), *m*/*z* 69.2 (intensity is 0.74×10^8), 85.1 (intensity is 0.09×10^8), 115.2 (intensity is 1.08×10^8), 143.2 (intensity is 0.31×10^8).

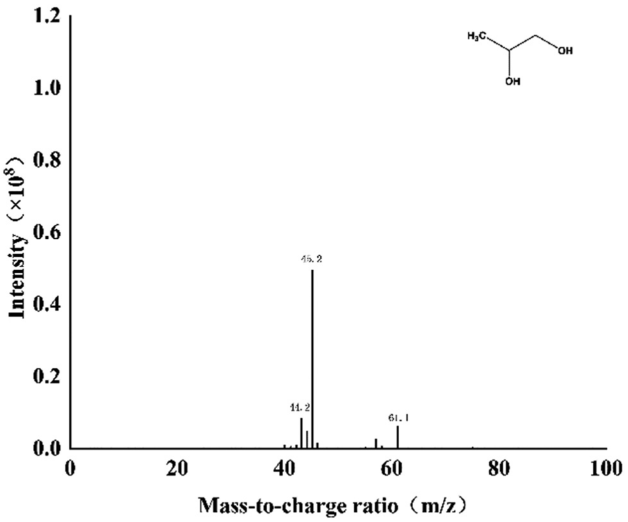


Figure 6: Bar graph of 1,2-propanediol glycol. (5) 1,2-Propanediol glycol (the mass spectral library matching score is 87.35), *m/z* 45.2 (intensity is 0.50 × 10^8), 61.1 (intensity is 0.06 × 10^8).

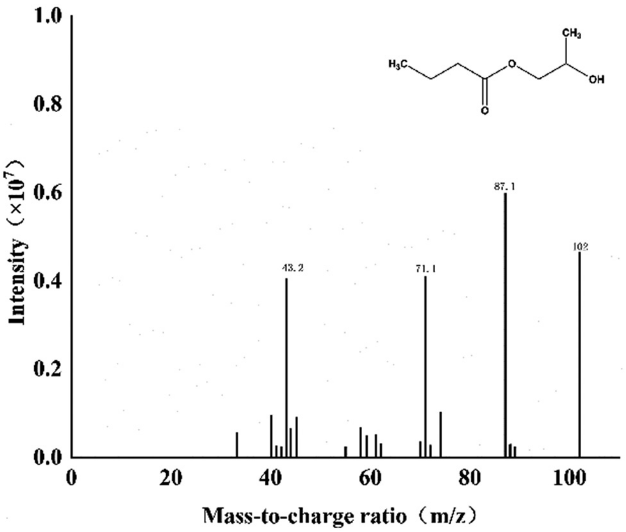


Figure 8: Bar graph of propane-1,2-diol 1-butyrate. (6) Propane-1,2-diol 1-butyrate (the mass spectral library matching score is 89.62), *m/z* 43.2 (intensity is 0.41 × 10^7), 71.1 (intensity is 0.41 × 10^7), 87.1 (intensity is 0.60 × 10^7), 102.1 (intensity is 0.47 × 10^7).

and alcohols. The specific classification of these by-products is provided in Table 2.

The reaction has produced several by-products, including heterocyclic compounds, esters, an acid, and alcohols. The heterocyclic compounds generated are 1,4-dioxane, 5,5-dimethyl-1,3-dioxane, and 5,5-dimethyl-1,3-dioxane-2-ethanol. The ester by-products are propanoic acid, 2-methyl-, 2-hydroxyethyl ester, isobutyric acid 2-ethyl-3-hydroxyhexyl ester, propane-1,2-diol, 1-butyrate, and isobutyl 2-ethylhexanoate. The acid by-product is propionic anhydride. The alcohol by-products generated are 1,2-propanediol glycol and neopentyl glycol.

4.3 By-product formation mechanisms

4.3.1 Formation mechanism of 1,4-dioxane

According to the catalytic mechanism of butyl tin tris (2-ethylhexanoate), the compound reacts with dibasic

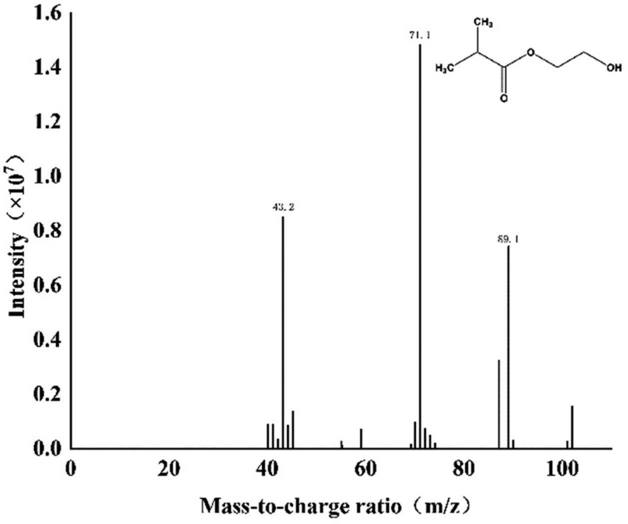


Figure 7: Bar graph of propanoic acid, 2-methyl-, 2-hydroxyethyl ester. (7) Propanoic acid, 2-methyl-, 2-hydroxyethyl ester (the mass spectral library matching score is 85.7), *m/z* 43.2 (intensity is 0.85 × 10^7), 71.1 (intensity is 1.49 × 10^7), 89.1 (intensity is 0.74 × 10^7).

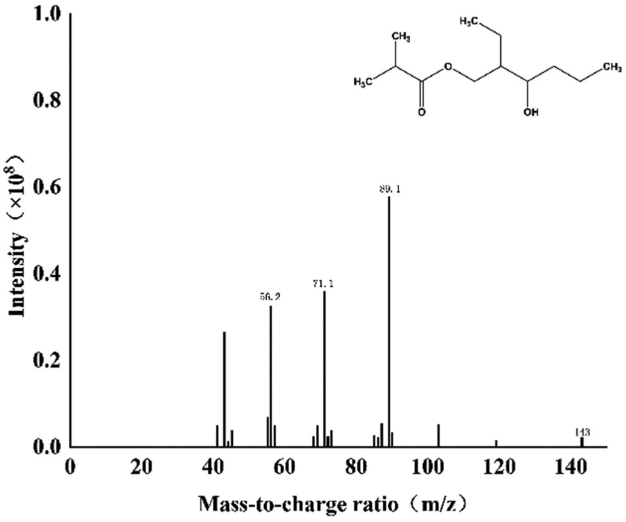


Figure 9: Bar graph of isobutyric acid 2-ethyl-3-hydroxyhexyl ester. (8) Isobutyric acid 2-ethyl-3-hydroxyhexyl ester (the mass spectral library matching score is 82.94), *m/z* 56.2 (intensity is 0.33 × 10^8), 71.1 (intensity is 0.36 × 10^8), 89.1 (intensity is 0.58 × 10^8), 143.2 (intensity is 0.02 × 10^8).

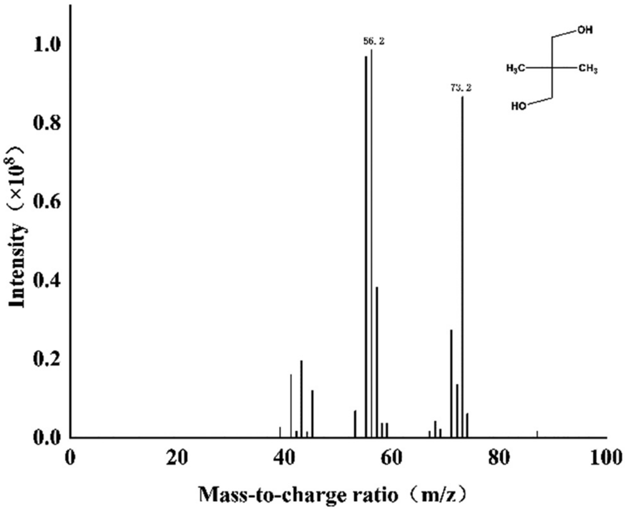


Figure 10: Bar graph of neopentyl glycol. (9) Neopentyl glycol (the mass spectral library matching score is 95.68), *m*/*z* 56.2 (intensity is 0.99×10^8), 73.2 (intensity is 0.87×10^8).

acid, leading to the breaking of the Sn–OX bond in butyl tin tris (2-ethylhexanoate) and the O–H bond in the dibasic acid. In the first step of the reaction, H–OX is produced, which then releases H^+ and creates acidic conditions that promote the cyclization reaction of diethylene glycol (15) (Figure 15).

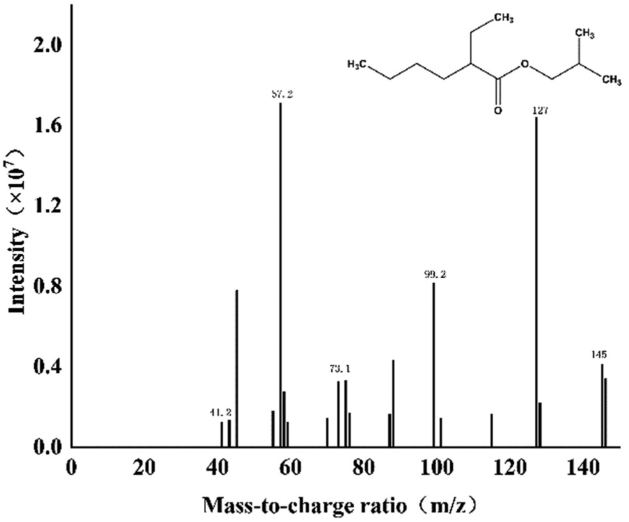


Figure 11: Bar graph of isobutyl 2-ethylhexanoate. (10) Isobutyl 2-ethylhexanoate (the mass spectral library matching score is 91.58), *m*/*z* 41.2 (intensity is 0.12×10^7), 57.2 (intensity is 1.71×10^7), 73.1 (intensity is 0.33×10^7), 99.2 (intensity is 0.81×10^7), 127.2 (intensity is 1.64×10^7), 145.2 (intensity is 0.42×10^7).

4.3.2 Formation mechanism of 5,5-dimethyl-1,3-dioxane-2-ethanol

4.3.2.1 Step one

The first step of the reaction produces HOX, which releases H^+ and creates an acidic environment. This acidic

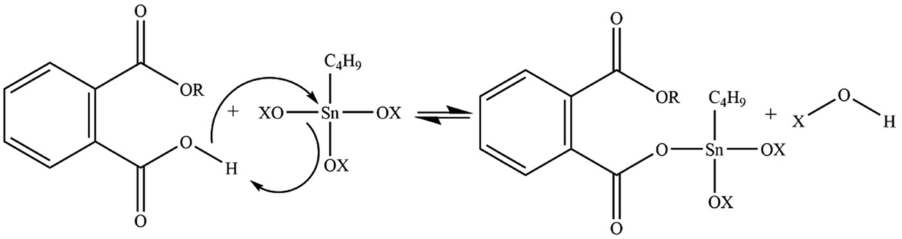


Figure 12: Catalytic mechanism of butyl tin tris (2-ethyl hexanoate).

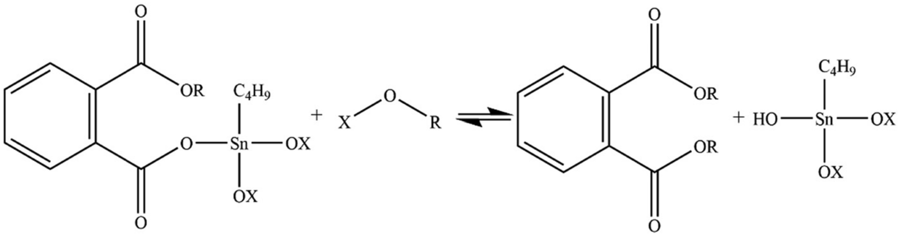


Figure 13: Catalytic mechanism of butyl tin tris (2-ethyl hexanoate).

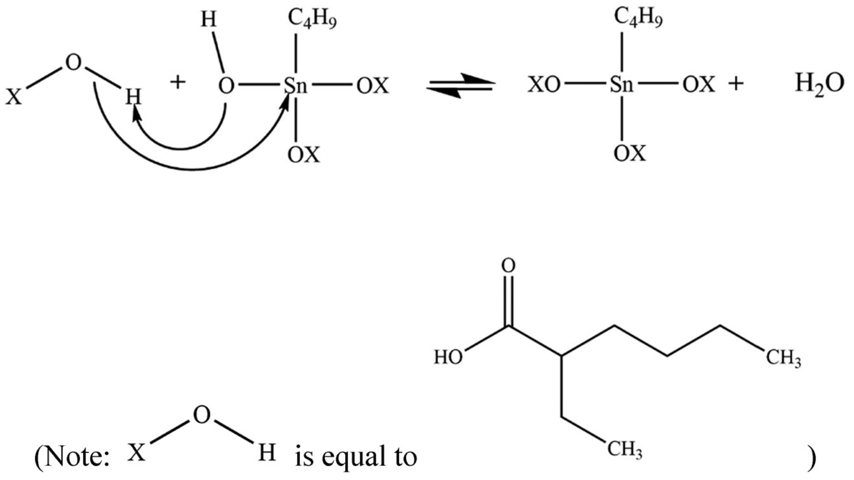


Figure 14: Catalytic mechanism of butyl tin tris (2-ethyl hexanoate).

Table 2: Classification of by-products

Number	Classification	By-products
I	Heterocyclic compounds	1,4-Dioxane
II		5, 5-Dimethyl-1,3-dioxane
III		5,5-Dimethyl-1,3-dioxane-2-ethanol
IV	Esters	Propanoic acid,2-methyl-,2-hydroxyethyl ester
V		Isobutyric acid 2-ethyl-3-hydroxyhexyl ester
VI		Propane-1,2-diol 1-butyrate
VII		Isobutyl 2-ethylhexanoate
VIII	Acids	Propionic anhydride
IX	Alcohols	1,2-Propanediol glycol
X		Neopentyl glycol

environment is crucial for the elimination reaction to occur. It causes 1,2-propanediol glycol to undergo shedding of OH at the No. 2 position and H on the neighboring carbon, resulting in the formation of a molecule of H_2O and allyl alcohol (16). The allyl alcohol then undergoes an addition reaction in the presence of a metal-tin compound to form 1,3-propanediol (Figure 16).

4.3.2.2 Step two

Under the action of a catalyst, one hydroxyl group of 1,3-propanediol is oxidized to a carbonyl group. In an acidic environment, butyl tin tris (2-ethyl hexanoate) induces a positive charge on the carbonyl carbon in the aldehyde, while the hydroxyl group in neopentyl glycol carries a negative charge. The positively charged carbonyl carbon and the negatively charged hydroxyl group undergo a nucleophilic addition reaction, resulting in a temporary ester intermediate. A proton transfer occurs between the carbonyl oxygen and the hydroxyl oxygen inside the ester intermediate, generating the cyclic product 5,5-dimethyl-1,3-dioxane-2-ethanol and one molecule of water (17) (Figure 17).

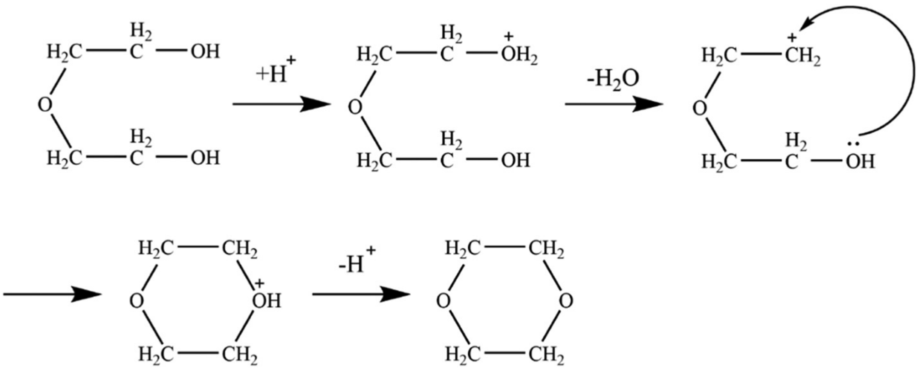


Figure 15: Formation mechanism of 1,4-dioxane.

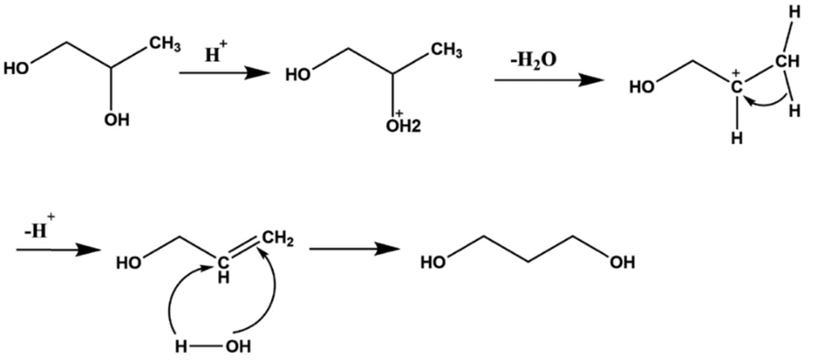


Figure 16: Formation mechanism of 1,3-propanediol.

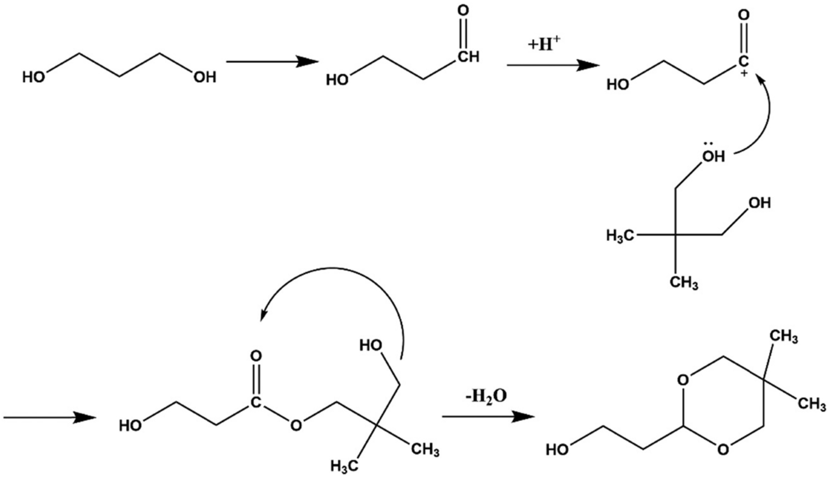


Figure 17: Formation mechanism of 5,5-dimethyl-1,3-dioxane-2-ethanol.

4.3.3 Formation mechanism of 5,5-dimethyl-1,3-dioxane

In the presence of a catalyst, 1,3-propanediol is thermally cracked with formaldehyde as the main product. In an acidic environment, butyl tin tris (2-ethyl hexanoate) causes a positive charge on the carbonyl carbon in formaldehyde, while the hydroxyl group in neopentyl glycol carries a negative charge. The reaction proceeds via a

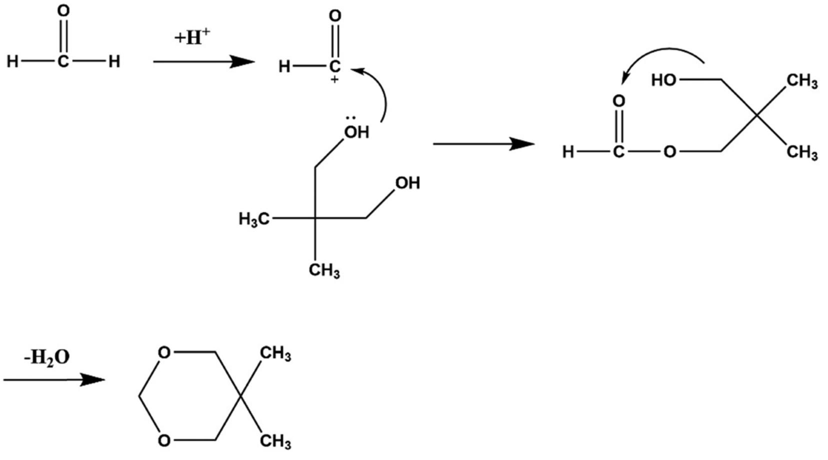


Figure 18: Formation mechanism of 5,5-dimethyl-1,3-dioxane.

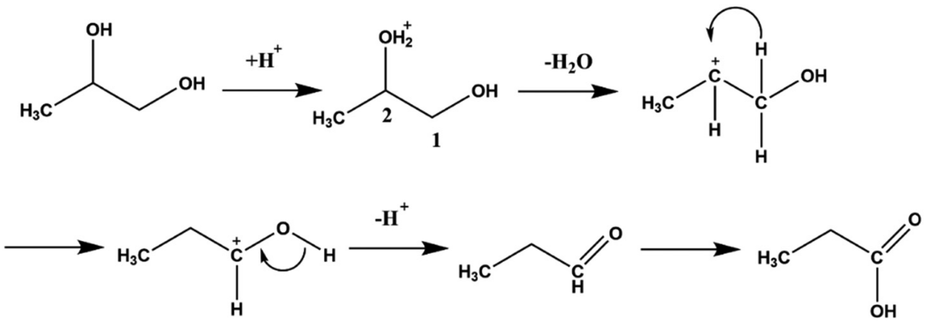


Figure 19: Formation mechanism of propionic acid.

nucleophilic addition between the positively charged carbonyl carbon and the negatively charged hydroxyl group, generating a transient ester intermediate. Subsequently, a proton transfer occurs between the carbonyl oxygen and the hydroxyl oxygen within the ester intermediate, leading to the formation of the cyclic product 5,5-dimethyl-1,3-dioxane and the liberation of one molecule of water (Figure 18).

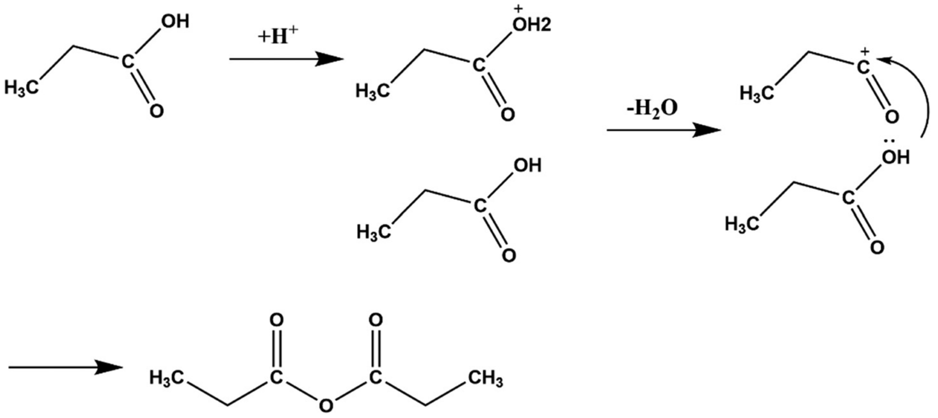
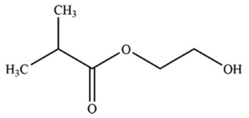
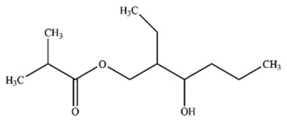
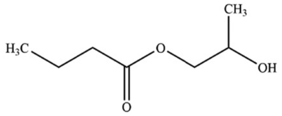
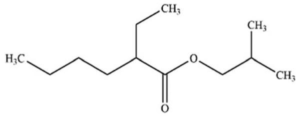


Figure 20: Formation mechanism of propionic anhydride.

Table 3: Structural formula of esters

Classification	By-products	Structure
Esters	Propanoic acid,2-methyl-,2-hydroxyethyl ester	
	Isobutyric acid 2-ethyl-3-hydroxyhexyl ester	
	Propane-1,2-diol 1-butyrate	
	Isobutyl 2-ethylhexanoate	

4.3.4 Formation mechanism of propionic anhydride

4.3.4.1 Step one

The first step of the reaction produces HOX, which releases H^+ and creates acidic conditions. Under these acidic conditions, the hydroxyl group at the No. 2 position of 1,2-propanediol reacts with the H^+ to form a protonated hydroxyl group. The protonated hydroxyl group undergoes dehydration, leading to the shedding of one molecule of H_2O. The hydrogen atom on the neighboring carbon (No. 1 position) is then removed, resulting in the formation of propionaldehyde (18). Propionaldehyde is immediately oxidized by the tin-metal compound to produce propionic acid (Figure 19).

4.3.4.2 Step two

Under acidic conditions, an additional H^+ is introduced to the system, causing propionic acid to undergo heating and dehydration. This process leads to the formation of propionic anhydride (19) (Figure 20).

4.3.5 Formation mechanism of esters

Under the conditions of butyl tin tris(2-ethylhexanoate)-catalyzed ester exchange, the intermediate product of butyl tin tris(2-ethylhexanoate) provides H^+, which initially react with the carbonyl group of the primary ester to form a carbon-cation intermediate. The alcohol molecules, which have a high affinity for protons, then combine with the carbon cation to form an intermediate product. Subsequently, this intermediate product decomposes, producing H^+ to catalyze the next round of reactions. These processes lead to the formation of the esters given in Table 3.

4.3.6 Formation mechanism of alcohol

1,2-Propanediol glycol and neopentyl glycol are raw materials carried out by water.

5 Conclusion

1. In this experiment, we used butyl tin tris (2-ethylhexanoate) as a catalyst to synthesize a 50/50 polyester/epoxy powder coating. During the process, we analyzed the by-products using GC–MS and identified different organic compounds that were classified into four categories: cyclic, ester, acid, and alcohol. The cyclic by-products included 1,4-dioxane, 5,5-dimethyl-1,3-dioxane, and 5,5-dimethyl-1,3-dioxane-2-ethanol. The ester by-products were propanoic acid, 2-methyl-, 2-hydroxyethyl ester, isobutyric acid 2-ethyl-3-hydroxyhexyl ester, propane-1,2-diol, 1-butyrate, and isobutyl 2-ethylhexanoate. The acid by-product was propionic anhydride, and the alcohol by-products were 1,2-propanediol glycol and neopentyl glycol.
2. The catalytic process of butyl tin tris (2-ethylhexanoate) forms 2-ethylcaproic acid, which creates an acidic environment that facilitates the cyclization reaction. The cyclization of diethylene glycol was observed to occur easily under its catalysis, indicating the need for careful attention to this phenomenon.
3. It is important for the factory to pay attention to the production of propionic anhydride since it emits an irritating odor. Therefore, proper ventilation and personal protective equipment should be used to prevent exposure and ensure the safety of workers. Additionally, measures should be taken to minimize the formation of propionic anhydride during the reaction process. Regular monitoring of the reaction products and waste streams is also important to ensure compliance with safety and environmental regulations.

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