# Preparation and Application of Phosphorus-Nitrogen Synergistic Flame Retardant Polyester Polyols

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**Abstract:** In this paper, N,2- hydroxyethyl, FRC-6, hexanediol and so on were used as raw materials to synthesize and design a new type of JZP, to detect its structure by infrared spectroscopy and nuclear magnetic resonance, and to use it as a chain extender for soft chain segments, and to react with both PPG1000 and TDI, so as to prepare coating materials except TPU. this material has many characteristics. testing this material, it can be seen that the increase of JZP content has an increasing effect on the average particle size of the TPU emulsion, its viscosity is reduced and its water resistance is enhanced. at the same time, the effect of JPZ addition on the TPU is also huge, which can improve the flame retardancy of the material very well, and the application effect of the material is good.

## 1. Introduction

As an environmentally friendly polymer material, waterborne polyurethane has many advantages, first of all, it uses water as a solvent, so it can ensure no toxicity and no pollution to the environment. As a coating material, it can be widely used in a variety of coatings, such as furniture surface coatings, textile coatings and so on. But it also has some drawbacks, this material is easy to burn, so its scope of application and development prospects have been limited. In recent years, with the rise of environmental protection materials [1]. The research of flame retardant waterborne polyurethane materials is on the agenda, especially the flame retardant and wake-up polyurethane materials which can replace the traditional flame retardant are popular. Flame retardant waterborne polyurethane was prepared by OP550 and hydroxyl dinitrohexyl as well as OP550 and silicone polyols. OP550 is a product containing phosphorus polyether polyols, which can increase the flame retardant effect in the reaction of polyurethane, but because it is imported from Germany, it is expensive and the cost of preparation is high, so the comprehensive effect of use is not good.

This paper proposes another way of thinking, N,2- hydroxyethyl, FRC-6, hexanediol and so on as raw materials, synthesis and design into a new JZP.[2]. The JZP was used as a soft satin chain extender to give fire-retardant effect to wake-up polyurethane coating, and the TPU coating material was prepared. various properties of TPU materials are tested and studied in this paper.

## 2. Experimental Experiments

### 2.1. Raw Materials and Reagents

The raw materials for this test are adipic acid (Adipic acid,AA), N ,2-hydroxyethyl, FRC-6, DBTDL, HDO, TDI, DMPA and PPG1000..

### 2.2. Synthesis of Phosphorous Polyester Diols

Add FRC-6 The catalyst mol, 0.7 mol, hexanediol 0.8 and adipic acid 1 mol were added into

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three flasks with thermometers, agitators and condensing tubes respectively.$^{o}$C make it undergo esterification reaction. Remove the vacuum and remove water after 30 minutes of esterification. Repeat the process until the reaction is over [3]. The acid value in the reaction is tested and the material is discharged after the reaction is finished. The raw acid value reached 20 mgKOHG/g and the hydroxyl value was 140 mg KOH/g. the polyester synthesis route is shown in figure 1.

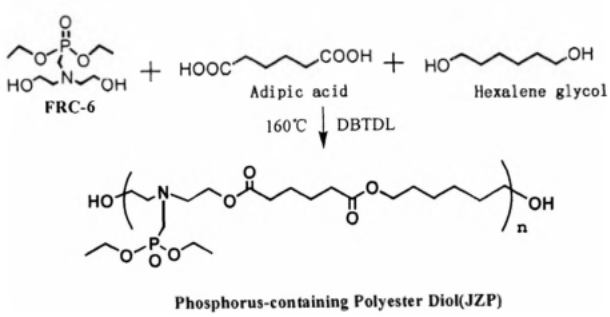


Figure 1 Polyester synthesis route

### 2.3. JZP Flame Retardant Modified Waterborne Polyurethane Synthesis of Soft Satin

The R values of - NCO and - OH selected in this paper are 1.6, DMPA 6 percent of the total content of the station, Add 5%,10%,15%,20% and 25% of the total JZP solid content to calculate the formula, And according to the results of this synthesis experiment, naming the samples obtained at each stage as TPU1, 2, 3, 4, 5. synthesis methods as follows, The JZP and PPG1000 and TD were added to four bottles at different stages, Four bottles are equipped with a thermometer, a reflux condenser tube and a stirrer. To stir and rise to 85$^{o}$C, and react at this temperature for up to 2 hours, then add DMPA, to reflect that the NCO content reaches the theoretical value, cool it down to 45$^{o}$C, add triethylamine and neutralize it for 30 minutes before adding butanone [4]. TPU and prepolymer were TPU and then emulsified in cold water for 10-20 minutes. The emulsion obtained after emulsification was set still for 10 hours. After that, butanone was removed by spinning and the TPU emulsion was obtained.

## 3. Characteristics of Phosphorus-Nitrogen Synergistic Flame Retardant

Phosphorus-nitrogen co-effect flame retardant has the characteristics of both phosphorous flame retardant and nitrogen flame retardant. It has the functions of flame retardant, heat insulation and oxygen insulation. The flame retardant principle of phosphorus-nitrogen co-effect flame retardant is through the co-action of condensed phase flame retardant and gas phase flame retardant, which shows that the flame retardant is classified as inorganic acid by decomposing heat absorption, which is the formation of protective film on the surface, so that the external air can be isolated. At the same time after thermal combustion, this flame retardant will release nitrogen, water vapor and other flame retardant gases, these gases cut off the oxygen in the air, and with the thermal action of flame retardants, reduce most of the temperature. at the same time, the matter of phosphorus oxides and nitrogen compounds forming the coking carbon structure is covered in the remaining carbon, which interrupts the effect of combustion and thus inhibits combustion. Phosphorus-nitrogen co-effect flame retardant has many characteristics, first of all, it is less harmful to the environment, and can play a good role in flame retardant, and second, it has the characteristics of phosphorus-nitrogen flame retardant. It has good flame retardancy and has synergistic flame retardancy with nitrogen compounds. In addition, it has good stability, non-toxic and other advantages.

## 4. Application of Phosphorus-Nitrogen Synergistic Flame Retardant

Phosphorus-nitrogen synergistic flame retardants are mainly divided into two types, one is reactive [5]. One is additive. Most of the current use belongs to the additive type, mainly including the expansion type flame retardant, other types of flame retardant and so on. The intumescent flame

retardants are the most important flame retardants, which are widely used. PNC is the core element, through the proportion to form a certain amount of carbon, dehydrating agent and gas source. The new nitrogen-containing phosphate esters are mainly synthesized in the research of intumescent flame retardants. Phosphonitrile flame retardants are also one of the common types, which are inorganic organic compounds formed by PN as the most important framework. In recent years, the flame retardants as the main research direction has become the main development trend, received attention from all walks of life. It not only has good flame retardancy, but also has synergistic flame retardancy with nitrogen compounds. In addition, it has good stability, non-toxic and other advantages. In addition, it contains nitrogen, oxygen and phenanthrene flame retardants, which have many advantages, high phosphorus content, good heat resistance, and water resistance is not volatile. The application of the flame retardant with phosphorus-nitrogen co-effect synthesized by this flame retardant is very good. At present, there are many countries in this field that have mature research.

The non-toxic and halogen-free flame retardants have become an important research feature in the current research on flame retardants. The traditional flame retardants containing a single flame retardant element can not meet the needs of various fields. The development of flame retardants requires many flame retardants. However, there are many problems in this field, such as the effective flame-retardant effect when used alone, the need to mix the corresponding substances to increase the flame-retardant effect, and because the amount of flame-retardant will affect the mechanical properties of the basic materials, the two are incompatible.

## 5. Test Results

For the performance of the TPU emulsion synthesized in this experiment, the following are the specific results.

### 5.1. TPU Size Distribution of Emulsions

The specific situation of different sample size distribution can be seen from figure 2. it can be found that the particle size of TPU emulsion with different JZP content is mainly a single peak trend, which can show that the particle size distribution is more uniform and can improve the stability of the whole emulsion. With the addition of JZP, the average particle size of emulsion increases gradually, but the average is about 60 nm, which indicates that the particle size distribution of emulsion is more uniform. At the same time, after testing, it was found that the circular latex samples were mainly present but not agglomerated, so it can be concluded that the dispersion of emulsion is relatively stable.

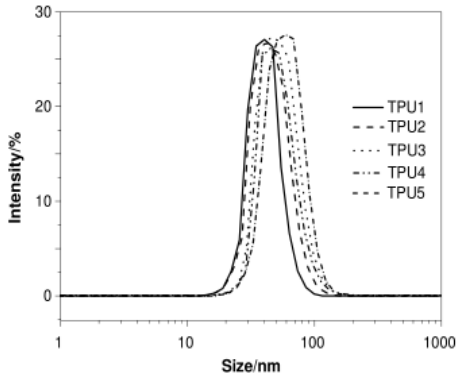


Figure 2 Preparation

### 5.2. TPU Flame Retardancy and Carbon Residue of Emulsion

The following figure shows the test results of the flame retardancy and carbon residue rate of the samples obtained from different stages. It can be seen that when the JZP addition amount is increased, the carbon residue rate of the samples appears to increase, in which the L0I value of different TPU is greater than 28%, which can indicate that all samples have flame retardancy effect

branch number, TPU have many advantages, its phosphorus content is high, it has good heat resistance, and the water resistance number is not easy to volatilize. while all the samples in the UL-94 reached the optimal grade, and the flame retardant effect was excellent, which was due to the addition of JZP to the polyurethane molecular chain, which added many PN structures. From the above, it can be seen that the flame retardant effect of the PN structure is remarkable, and a protective film will be formed in the material combustion chamber to protect the material to reduce the temperature, in addition, it can insulate the external air, so as to better play the effect of flame retardant. Due to the presence of nitrogen elements, many other flame retardants will be produced, which will increase the flame retardancy effect.

Tab.2 Flame retardant data of TPU samples

| Samples | WPU | TPU1 | TPU2 | TPU3 | TPU4 | TPU5 |
|---|---|---|---|---|---|---|
| Char yield% | 3.1 | 4.4 | 6.5 | 11.8 | 12.1 | 12.9 |
| LOI% | 18 | 28.7 | 29.0 | 29.5 | 29.1 | 28.6 |
| UL-94 | - | V-1 | V-0 | V-0 | V-0 | V-0 |

Figure 3 Preparation

## 6. Summary

To sum up, the TPU emulsion with PN synergistic structural steel synthesized in this paper has the characteristics of uniform dispersion of latex examples, its water absorption is low and water resistance is good, and it is found that the flame retardant effect is very remarkable in the flame retardant experiment, and the preparation cost of this material is low, the process is relatively simple, and it also has the characteristics of non-toxic and green environmental protection. Therefore, such materials are very suitable for use as coating materials, in the future such materials are very promising. At present, there are many countries in this field that have mature research, because of the late research time and short period, the development space of our country is very large.

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