

# Preparation and Microscopic Analysis of Polyurethane Elastomer Made by *Salix psammophila*

Jin-tian HUANG<sup>1, a</sup>, Ya-bin LI<sup>1, b</sup>, Kun-ling TIE<sup>1, c</sup>

<sup>1</sup>College of Material Science and Art Design, Inner Mongolia Agricultural University, Hohhot, China

<sup>a</sup>email:jintian\_h@163.com, <sup>b</sup>email:liyabin2258@163.com, <sup>c</sup>email:tiekunling7008@126.com

**Keywords:** the degradation of *Salix psammophila*; polyurethane elastomer; microscopic analysis

**Abstract:** In this paper, the degradation of *Salix psammophila* is used to make the polyurethane elastomer and study on the process involving changes in molecules with SPM. Compared with the pure polyurethane, it can be concluded that the polymerization of particles' size, height and measurement have got bigger visibly. But the cured particles change nothing and retain the natural polymer's linear permutation. And this polyurethane elastomer's fracture profile is brittle.

## Introduction

Wood materials are a plural system. Especially the network consisted of lignin, the crystalline structure of cellulose and hemi-cellulose is rich in carboxyl, carbonyl, phenol hydroxyl and hydroxyl<sup>[1]</sup>. And the activity mass can react with isocyanate to prepare polyurethane. Not only can it improve the utilization of resources, but also reduce the cost and the dependence of petroleum products for the polyurethane industry. It plays an important role in alleviating the environmental crisis and the energy pressure. Due to its excellent compatibility, it can be used to make homogeneous materials. The macro chain of this polyurethane will be broken by microbial, until absolute degradation<sup>[2, 3]</sup>

## Experimental

**material and reagent.** *Salix* material degradation solution : self-made<sup>[4]</sup>; Isocyanate(MDI) was chemical pure; MOCA was curing agent (the Xiangyuan chemical Co.Ltd of Suzhou); ethylene glycol, sulfuric acid and acetone were analytically pure .

Electronic analytical balance ( BS210S Beijing Sartorius electronics Co.Ltd), vacuum drying oven (DZF-6210 Shanghai yiheng instruments Co. Ltd),blast oven (101A-3B Shanghai Anxiang scientific instruments Co.Ltd), water ring vacuum pump ( SHZ-D(III) Shanghai Shengguang instruments Co.Ltd) ,heating mixer (Jinan Junsheng testing instrument equipment Co.Ltd).

**Determination of initial hydroxyl.** Test method: Phthalic anhydride-Pyridine<sup>[5]</sup>

**The process of polyurethane's preparation.** Firstly, the degradation of *Salix psammophila* was put into a three-neck flask, then dehydration for 1.5-2h, keep the negative pressure 0.086Mpa and the temperature 100-120°C. When temperature fell to 60°C, put certain melted MDI into the three-neck flask, reacted 1.5h at 70±2°C. Then degassing the reactants, after cool down, stored in a bottle before use. The prepolymer were named A. Secondly, a certain amount of mixture of degradation and MOCA was selected, which was heated to 100~120°C. After dehydration, put catalyst into it. The mixture were named B. Finally, A and B were fixed in proportion and poured into mold, cured it at 120°C. After 1 h, the samples were tested.

## Microscopic analysis of polyurethane elastomer

**Application of SPM in polyurethane elastomer.** The sample was analyzed with atomic force microscope (CSPM550) manufactured by HTTACH, Japan, the mode was contact and N, N-dimethyl formamide as disperser.

**Analysis by scanning electron microscope.** The profile of sample was analyzed by scanning electron microscope (S-3400N) manufactured by HTTACH, Japan.

**Results and Conclusions**

**Analysis of polyurethane elastomer by scanning probe microscope**

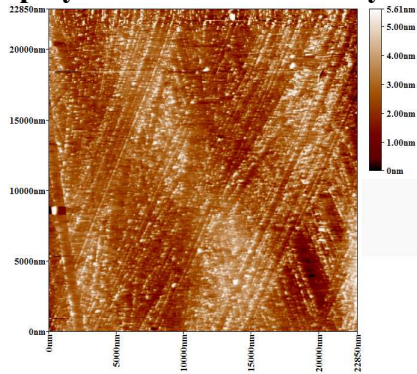


Fig.1 SEM image of degradation of *Salix psammophila*

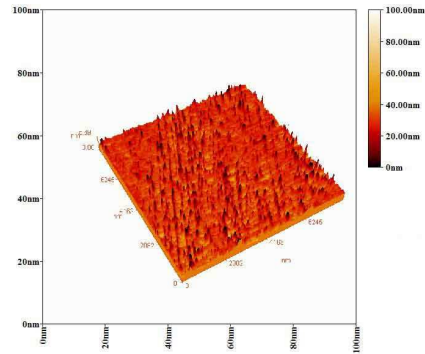


Fig. 2 3D-SEM image of degradation of *Salix psammophila*

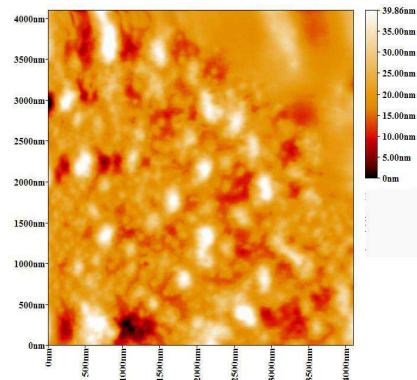


Fig. 3 SEM image of prepolymer

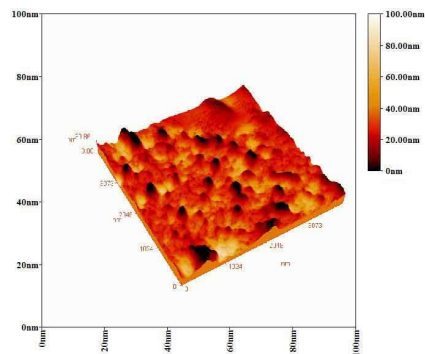


Fig. 4 3D- SEM image of prepolymer

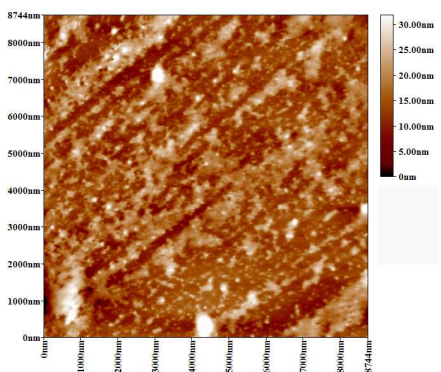


Fig.5 SEM image of polyurethane elastomer

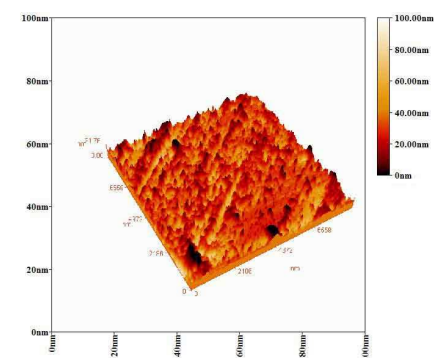


Fig.6 3D-SEM image of polyurethane elastomer

Table 1 Particle size analysis by scanning electron microscope

	average particle size	≤10%	≤50%	≤90%
Degradation of <i>Salix psammophila</i>	0.93[nm]	0.40[nm]	0.83[nm]	1.60[nm]
Prepolymer of polyurethane elastomer	1.03[nm]	0.40[nm]	0.80[nm]	1.70[nm]
polyurethane elastomer	1.02[nm]	0.40[nm]	0.80[nm]	1.70[nm]

Table 2 Height analysis by scanning electron microscope

	average height	≤10%	≤50%	≤90%
Degradation of <i>Salix psammophila</i>	80.337[nm]	70.000[nm]	75.000[nm]	90.000[nm]
Prepolymer of polyurethane elastomer	77.619[nm]	60.000[nm]	75.000[nm]	85.000[nm]
polyurethane elastomer	78.357[nm]	65.000[nm]	75.000[nm]	90.000[nm]

Table 3 Area analysis by scanning electron microscope

	average area	maximum area	minimum area
Degradation of <i>Salix psammophila</i>	0.86[nm <sup>2</sup> ]	7.69 [nm <sup>2</sup> ]	0.04 [nm <sup>2</sup> ]
Prepolymer of polyurethane elastomer	1.23[nm <sup>2</sup> ]	52.33[nm <sup>2</sup> ]	0.04 [nm <sup>2</sup> ]
polyurethane elastomer	1.07[nm <sup>2</sup> ]	45.63[nm <sup>2</sup> ]	0.04 [nm <sup>2</sup> ]

From Fig.1 and Fig.2, we can see the large molecules of hemi-cellulose and cellulose in the degradation of wood materials are cracked into smaller molecules. And it has a narrower molecular weight distribution. The wood materials still retain the linear and regular permutation. From Table 1, Table 2 and Table 3 results show that the average particle size is 0.93nm, 50% of the average size is 0.83nm, the average height is 80.337nm, 50% of the average height is 75.000nm and the average area is 0.86nm<sup>2</sup>.

From Fig.3 and Fig.4, we can see the molecular orientation is in a condition of disarray after the reaction of isocyanate with degradation. Table.1 indicates that the average particle size of prepolymer is 1.03nm and it is 0.1nm bigger than that of the degradation and 50% the average particle size is 0.80nm. From the table 2, the average height is reduced to 77.619nm and 50% of the average height is 75.000nm. While the average area is 1.23nm<sup>2</sup> and it is 0.43nm<sup>2</sup> bigger than the degradation's. The reason is that the woody materials retain the stereo-chemical structure of macromolecular chains. And the activity mass of the branched chain will react with isocyanate. Thus the average height is decreased and the average is increased.

From Fig.5 and Fig.6, it shows that the molecular orientation of vulcanized polyurethane elastomer is tending regular. And from Table 1, the average particle changes little, however, from Table 2 and Table 3, the average height of the molecular increases 0.742nm and the average area of the molecular decreases 0.16nm<sup>2</sup>. Because there are many polar groups in the degeneration, the distance of intermolecular reduces, the hard section strengthens mutually, the ability of hydrogen bond formed is enhanced; Moreover with the content of hard section increasing, the length of hard section sequence also increases, and the micro-zone of hard segments form gradually, the tendency of ordered structure intermolecular in hard section is strengthen gradually. That leads to retain the natural material member line arrangement after sulfuration.

### The analysis of polyurethane elastomer by scanning electron microscope

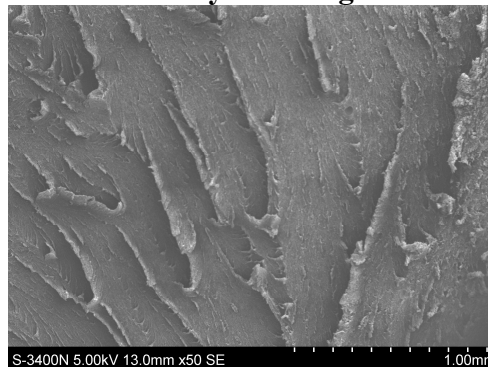


Fig 7 Profile of the polyurethane elastomer

Fig. 7 show the typical fracture, there are many stairs on the fracture surface, the stair converges mutually to form the “rivers” pattern. This is typical brittle fracture<sup>[6]</sup>, because the isocyanate is added firstly in the synthesis polyurethane elastomer process, due to the isocyanate acts as the hard section in the synthesis polyurethane process, simultaneously the content of isocyanate adjusts the hardness of polyurethane elastomer directly.

## Conclusions

After analysis the process of the polyurethane elastomer by scanning electron microscope, we can see the particle size of the cured elastomer changed little, while the height of particle increased, the average area decreased and the distance between the max area and min area also decreased. These polyurethane elastomers retain the natural polymer's linear permutation and have the similar properties with the degradation. The study provided a theory gist for getting better properties of polyurethane elastomer. Meanwhile, this polyurethane elastomer's fracture profile is brittle.

## Acknowledgement

This work was sponsored by the Applied Technology Research and Development in Inner Mongolia Science and Technology Department Project (20090514)

## References

- [1] Yoshida H, Morck R, Kringstad K P, et al. Kraft lignin in polyurethanes: Effects of the molecular weight of kraft lignin on the properties of polyurethanes from a kraft lignin- polymeric MDI system. *J Appl Polym Sci*, 1990, 40(11~12): 1819~1832.
- [2] Brdhimi B, Riedl B. Analysis of Polybutadiene grafted lignin material[C]. ANTEC Conference Proceedings, 1995, 3(2):2576—2581.
- [3] Yu-cang Zhang, ATSUSHI I, NARUHITO H, et al. Characterization of liquefied product from cellulose with phenol in the presence of sulfuric Acid[J]. *Bioresource Technology*, 2006, 97: 313-32
- [4] Li-ping Jin, Ya-bin Li, Xin Wang, Jin-tian Huang, A preliminary study on the degradation of *Salix psammophila* with hexylene glycol, college journal of Inner Mongolia agricultural university 2009 {2}, In Chinese
- [5] Ming-yuan Fu, application of polyurethane elastomer, {m} Presentation in CIAE, 199.6:286-289, In Chinese
- [6] Yu Zhou, analysis methods of materials, ( second edition) Machine Press, 2004, 215-237, In Chinese