Optimization of polyvinyl butyral synthesis process using response surface methodology and artificial neural network

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S1. Synthesis of PVB

The preparation procedure of PVB was as follows. First, PVA (5 g) was dissolved in deionized water ($w_{PVA} = 4\%\sim10\%$) vigorously stirring at about 85 °C for 1h until the solution was clear. Then, a small amount of SDS ($m_{SDS}/m_{PVA} = 0.01$) was added to the resulting PVA solution and maintained the temperature for 0.5 h. Next, the solution was cooled down to 60 °C, and a certain amount of catalyst was added to the solution and stirred for 0.5 h. After cooling down to 15 °C, n-butanal was added to it in drops within 0.5 h, and stirred for 2 h at 15 °C. Finally, the resulting mixture was slowly raised to the aging temperature and kept stirred for 2 h. After cooling down to room temperature, the obtained PVB products were filtered and washed with water, then dried in a vacuum at 50 °C.

S2. Preparation and selection of DES

The DESs were prepared as follows: a certain amount of HBD and HBA were mixed at about 60~65 °C with constant stirring until homogeneous and transparent liquids were formed. After cooling to room temperature, the obtained DESs were sealed and placed in a desiccator for experimental use.

Three DESs consisting of dodecyl trimethyl ammonium chloride (DTAC) and p-toluene sulphonic acid (PTSA), benzyltrimethylammonium chloride (BAC) and PTSA, and BAC and oxalic acid (OA), respectively, were prepared and utilized as catalysts for PVB synthesis. Experiments of selecting catalysts were carried out according to the procedures of PVB synthesis described above, in which the addition of DES was same of $m_{\text{DES}}/m_{\text{PVA}} = 0.20$, and the AD values are listed as Table S1. The results showed that BAC-PTAC exhibited the best catalytic activity. The molar ratio of BAC and PTAC was investigated on the catalytic effect of DES the in our previous work, and results showed that PVB products prepared by BAC-PTSA had smaller and more uniform particle sizes. Therefore, BAC-PTSA is more suitable for catalyzing the synthesis of PVB, and it was selected as the catalyst for PVB synthesis in this study, and it was selected as the catalyst in this study.

Table S1 The values AD of PVB catalyzed by three types of DESs

Туре	AD (%)
DTAC-PTSA	75.36%
BAC-PTSA	80.32%
BAC-OA	77.83%

S3. Characterization of DES

The FTIR spectrum of PTSA, BAC, and BAC-PTSA is shown in Figure S1. As shown in Figure S1, DES has similar peaks to the pure components. The formation of hydrogen bonds after the combination of BAC with PTSA resulted in changes in the spectrum, more specifically broadening or shifting of the bands. In the DES spectrum,

the peaks at about 1162 cm⁻¹ and 1120 cm⁻¹ are attributed to the asymmetric stretching bands of SO₃, while the peak at about 1002 cm⁻¹ belongs to the symmetric stretching vibration of SO₃. In addition, the peaks at about 3043 cm⁻¹ and 894 cm⁻¹ correspond to the symmetric and asymmetric stretching bands of N⁺(CH₃)₃, respectively.²⁻⁴

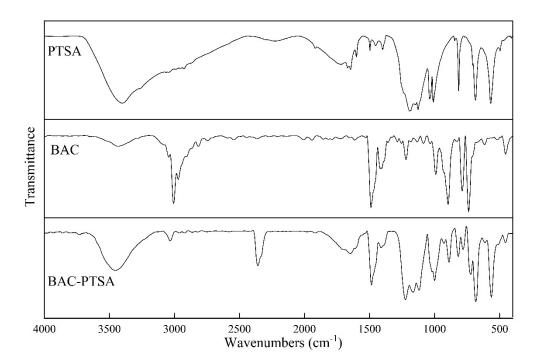


Figure S1. FTIR spectrum of PTSA, BAC and BAC-PTSA.

References

- 1. W. Luan, J. Xu, Z. Zeng, W. Xue and Y. Bai, Can. J. Chem. Eng., 2023, 1.
- 2. L. Tang, Y. Guo, J. Xu, Z. Zeng and W. Xue, *Braz. J. Chem. Eng.*, 2022, **39**, 715-726.
- 3. S. Jiang, Z. Zeng, W. Xue, W. Zhang and Z. Zhou, *Korean J. Chem. Eng.*, 2020, **37**, 1482-1489.
- 4. N. Rodriguez Rodriguez, L. Machiels and K. Binnemans, *ACS Sustain. Chem. Eng.*, 2019, 7, 3940-3948.