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GROUPS CHARACTERISTICS OF BIOACTIVATOR EXTRACTIVES IN THREE POPLAR WOODS

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ABSTRACT

Populus nigra, *Populus lasiocarpa* and *Populus tomentosa* were the dominant plantation tree species in China. Their extractives had the obvious biological activity, but could not be used richly collected from the Dongtinghu Forest Zone of Hunan Province, the Zhumadian Forest Zone of Henan Province, and the Linyi Forest Zone of Shandong Province, China, respectively. The result shown that the groups characteristics of three *populus* wood extractives were evident. Among the single extractions, the absorbance of peaks in *Populus nigra* wood extractives reached mostly at the ethanol/methanol extractives, minimally at the benzene/ethanol extractives, the absorbance of peaks were different in *Populus lasiocarpa* wood extractives, and the absorbance of peaks in *Populus tomentosa* wood extractives reached mostly at the benzene/ethanol extractives, minimally at the ethanol/methanol extractives. Among the sequential extractions, the absorbance of peaks from LD147, LD173 and LD181 extractives were the weakest, ones from LD154, LD171 and LD189 extractives were the strongest.

KEYWORDS: Groups; *Populus nigra*; *Populus lasiocarpa*; *Populus tomentosa*; wood extractives; FT-IR.

INTRODUCTION

Poplar, which was the most important plantation tree, was cultivated widely in the north and southern China. Poplar grew rapidly and was tall with trunks of up to 2.5 m diameter. Its cutting period was 5~8 to 12 years (Lihua et al. 2007). The bark on young trees was smooth and white to greenish or dark grey, on old trees it remained smooth in some species; the leaves were spirally arranged, and vary in shape from triangular to circular or lobed, and often turned bright gold to yellow before autumn; the flowers were mostly dioeciously and appeared in early spring before the leaves; the fruit was a two to four-calved dehiscent capsule, green to reddish-brown, mature in mid summer, containing numerous minute light brown seeds surrounded by tufts of long, soft, white hairs which aid wind dispersal (http://en.wikipedia.org/wiki/Populus). Now there were more than 7.0 million hm^2 of poplar plantation in China, ranking top one in the world (Shengzuo 2008). And poplar was a species of wood that was commonly used in wooden products and used quite often for more industrial purposes, such as the core of finer plywood or for crates and pallets (Chunquan et al. 2001; Lihua et al. 2007).

Poplar wood was a type of wood that was often classified both as a hardwood and a softwood. Poplar wood was also sometimes referred to as yellow poplar or whitewood. *Populus nigra*, which was native to Europe, Asia and Africa, had a wide distribution area (Zsuffa 1974; François et al. 1998). *Populus lasiocarpa* and *Populus tomentosa*, which were the dominant species of plantation in north and central China, were planted more than 2 Mha (Yoshinori and Eckhard 1976; Ningxia et al. 2012). The three poplar wood were the softwood and whitewood, and were rich in wood extractives. However, poplar wood extractives were studied less and unknown. What's more, the different extractions were different because each solvent had different dissolving ability. Therefore, the group characteristics of wood extractives were investigated and analyzed by FT-IR after *Populus nigra*, *Populus lasiocarpa* and *Populus tomentosa* wood were extraction and their wood extractives were obtained.

MATERIAL AND METHODS

Materials

Populus nigra wood, *Populus lasiocarpa* wood and *Populus tomentosa* wood were collected from the Dongtinghu Forest Zone of Hunan Province, the Zhumadian Forest Zone of Henan Province, and the Linyi Forest Zone of Shandong Province, China, respectively. The fresh wood were shaved, powdered and kept in vacuum, respectively. Acetic ether, methanol, benzene, petroleum ether and ethanol were chromatographic grade, preparing for the experiments. Cotton thread and cotton bag were both extracted by benzene/ethanol ($V_{ethanol}$ /$V_{benzene}$ = 2) solution for 12 h.

Experiment methods

Single extraction

Weighed 3 pieces of each powder, each was about 20 g (0.1 mg accuracy) and finally parceled into the cotton bag tied by the cotton thread, and signed. Extraction was carried out in 350 ml solvents by the Foss method for 7 hours. Solvents were ethanol/methanol ($V_{ethanol}$ / $V_{methanol}$ = 2), petroleum ether / acetic ether ($V_{petroleum\ ether}$ / $V_{acetic\ ether}$ = 2), and benzene/ethanol ($V_{ethanol}$ / $V_{benzene}$ = 2), respectively. Ethanol/methanol extraction, petroleum ether / acetic ether extraction, and benzene/ethanol extraction were done under the condition of 75, 90 and 95°C, respectively. After extraction, the extractives were obtained by evaporation at 60~70°C.

Tab. 1: The extractives and single extraction methods.

Extractives No.	Tree species	Extraction method
LD4	*Populus nigra*	benzene/ethanol extraction
LD5	*Populus nigra*	petroleum ether / acetic ether extraction
LD6	*Populus nigra*	ethanol/methanol extraction
LD10	*Populus lasiocarpa*	ethanol/methanol extraction
LD11	*Populus lasiocarpa*	petroleum ether / acetic ether extraction
LD12	*Populus lasiocarpa*	benzene/ethanol extraction
LD19	*Populus tomentosa*	ethanol/methanol extraction
LD20	*Populus tomentosa*	petroleum ether / acetic ether extraction
LD21	*Populus tomentosa*	benzene/ethanol extraction

Sequential extraction

Weighed 9 pieces of each powder, each was 20 g (1.0 mg accuracy), and finally parceled by using the cotton bag and tied by using cotton thread, and signed. According to Tab. 2, the sequential extraction were done, and the extractives were obtained, respectively.

Tab. 2: The extractives and sequential extraction methods.

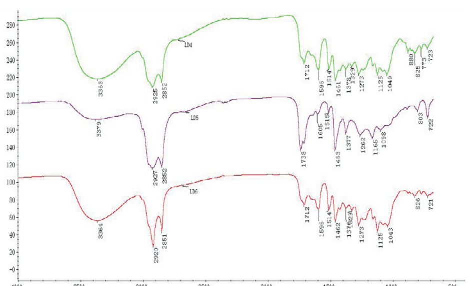
Extractives No.	Tree species	Step	Extraction method
LD146	*Populus lasiocarpa*	1st	benzene/ethanol extraction
LD151	*Populus lasiocarpa*	2nd	Ethanol/methanol extraction
LD153	*Populus lasiocarpa*	3rd	petroleum ether / acetic ether extraction
LD147	*Populus lasiocarpa*	1st	petroleum ether / acetic ether extraction
LD149	*Populus lasiocarpa*	2nd	benzene/ethanol extraction
LD152	*Populus lasiocarpa*	3rd	Ethanol/methanol extraction
LD148	*Populus lasiocarpa*	1st	Ethanol/methanol extraction
LD150	*Populus lasiocarpa*	2nd	petroleum ether / acetic ether extraction
LD154	*Populus lasiocarpa*	3rd	benzene/ethanol extraction
LD173	*Populus tomentosa*	1st	benzene/ethanol extraction
LD175	*Populus tomentosa*	2nd	Ethanol/methanol extraction
LD178	*Populus tomentosa*	3rd	petroleum ether / acetic ether extraction
LD174	*Populus tomentosa*	1st	Ethanol/methanol extraction
LD176	*Populus tomentosa*	2nd	petroleum ether / acetic ether extraction
LD180	*Populus tomentosa*	3rd	benzene/ethanol extraction
LD190	*Populus tomentosa*	1st	petroleum ether / acetic ether extraction
LD177	*Populus tomentosa*	2nd	benzene/ethanol extraction
LD179	*Populus tomentosa*	3rd	Ethanol/methanol extraction
LD182	*Populus nigra*	1st	benzene/ethanol extraction
LD186	*Populus nigra*	2nd	Ethanol/methanol extraction
LD188	*Populus nigra*	3rd	petroleum ether / acetic ether extraction
LD183	*Populus nigra*	1st	Ethanol/methanol extraction
LD184	*Populus nigra*	2nd	petroleum ether / acetic ether extraction
LD187	*Populus nigra*	3rd	benzene/ethanol extraction
LD181	*Populus nigra*	1st	petroleum ether / acetic ether extraction
LD185	*Populus nigra*	2nd	benzene/ethanol extraction
LD189	*Populus nigra*	3rd	Ethanol/methanol extraction

FTIR analysis

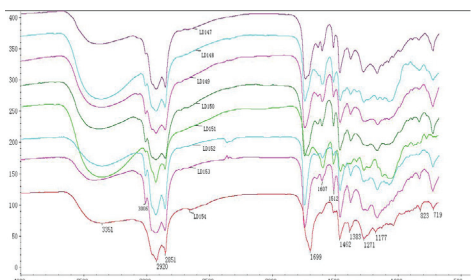
The extractives samples were recorded on a Thermo Nicolet FT-IR spectrometer (Thermo Fisher Nicolet, 670 FT-IR). Thirty-two scans were collected per sample at a spectral resolution of 4 cm^{-1}, and the collected spectra were normalized against air. The spectral range was from 4000 to 500 cm^{-1} (Wanxi et al. 2013; 2014a; b; Yong-Chang et al. 2014; Qiu et al. 2014; Lansheng et al. 2013a; b).

RESULTS AND DISCUSSION

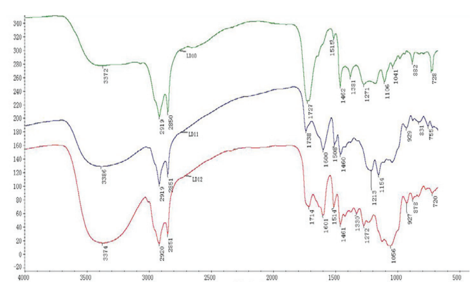
FT-IR spectrums of three wood extractives from *Populus nigra*, *Populus lasiocarpa* and *Populus tomentosa* were showed in Fig. 1 by single extraction. FT-IR spectrums of three wood extractives from *Populus nigra*, *Populus lasiocarpa* and *Populus tomentosa* were showed in Fig. 2 by sequential extraction. Based on FTIR spectra, bands at 3420, 2918, 1460, 1330, 1231, 1158, 1122, 897 cm^{-1} were assigned to O–H stretching in hydroxyl group, C-H stretching in methyl and methylene group, C-H deformation in methyl and methylene, syringyl, C-C plus C-O plus C=O stretching ($G_{condensed}$>$G_{etherified}$), C-O-C asymmetric stretching, Aromatic C-H (typical syringic aldehyde), polysaccharide *β*- bond stretching vibration. Characteristic peaks attributed to lignin were bands at 1593, 1504 and 1420 cm^{-1}. Their assignments were obtained according to the papers (Wanxi et al. 2013; 2014a; b; Yong-Chang et al. 2014; Qiu et al. 2014; Lansheng et al. 2013a; b).



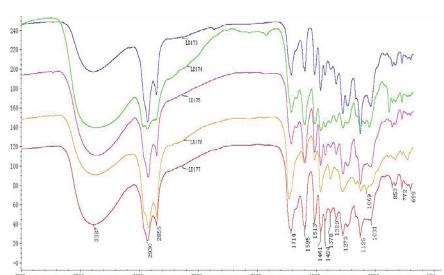
Populus nigra



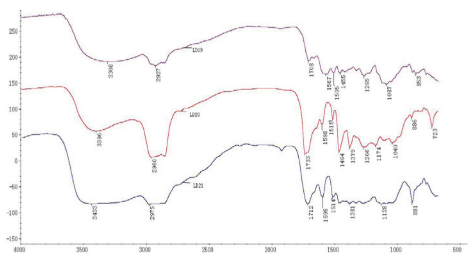
Populus lasiocarpa



Populus lasiocarpa

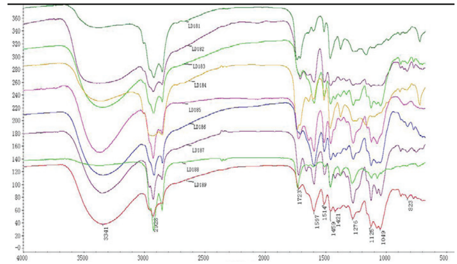


Populus tomentosa



Populus tomentosa

Fig. 1: FT-IR spectrums of three populus wood extractives of single extractions.



Populus nigra

Fig. 2: FT-IR spectrums of three populus wood extractives of sequential extractions.

Groups characteristics of three populus wood extractives of single extractions

FT-IR spectra of three populus wood extractives of single extractions were showed in Fig. 1. The most important O-H stretching in hydroxyl group was assigned to the 3308 - 3433 cm^{-1} region. At 3417 cm^{-1}, 1593, 1504, 1420, 1330, 1230 cm^{-1} were O-H stretching in hydroxyl group, aromatic skeletal vibration plus C=O stretching, aromatic skeletal vibrations, aromatic skeletal vibration combined with C-H in plane deformation, syringyl vibrations, benzene ring-hydrogen bond vibrations, respectively.

To wood extractives of *Populus nigra*, the absorbance of peaks at 3364~3379 cm^{-1} increased from 0.259 to 0.817. The absorbance of peaks at 1596~1605 cm^{-1} reduced from 0.570 to 0.161 at LD4, the absorbance of peaks at 1514 cm^{-1} reduced from 0.555 to 0.169 at LD4, and the absorbance of peaks at 1462 cm^{-1} reduced from 0.816 to 0.260 at LD4, resulting that the benzene ring of lignan reached the most destructive at LD4 extractives. The absorbance of peaks at 1377 cm^{-1} reduced from 0.565 to 0.156 at LD4 extractives, The absorbance of peaks at 1329 cm^{-1} reduced from 0.510 to 0.172 at LD4 extractives, the absorbance of peaks at 1262~1273 cm^{-1} reduced from 0.669 to 0.294 at LD4 extractives, the absorbance of peaks at 1125~1165 cm^{-1} reduced from 0.668 to 0.360 at LD4 extractives, the absorbance of peaks at 11043~11098 cm^{-1} reduced from 0.675 to 0.304 at LD4 extractives. The results shown that the absorbance of each peak reached mostly at the ethanol/methanol extractives, minimally at the benzene/ethanol extractives.

To wood extractives of *Populus lasiocarpa*, the absorbance of peaks at 3372~3386 cm^{-1} increased from 0.485 to 0.810. The absorbance of peaks at 1714~1738 cm^{-1} reduced from 0.887 to 0.162 at LD12, the absorbance of peaks at 1600 cm^{-1} reduced from 0.462 to 0.248 at LD12, and the absorbance of peaks at 1508~1515 cm^{-1} reduced from 0.426 to 0.160 at LD12. The absorbance of peaks at 1460 cm^{-1} reduced from 0.684 to 0.369 at LD12 extractives. The absorbance of peaks at 1330~1381 cm^{-1} reduced from 0.573 to 0.369 at LD12 extractives, the absorbance of peaks at 1272 cm^{-1} reduced from 0.650 to 0.384 at LD12 extractives, the absorbance of peaks at 1106~1154 cm^{-1} reduced from 0.936 to 0.624 at LD12 extractives. The results shown that the absorbance of each peak was different among the three wood extractives.

To wood extractives of *Populus tomentosa*, the absorbance of peaks at 3308~3433 cm^{-1} increased from 1.358 to 0.000. The absorbance of peaks at 1567~1598 cm^{-1} reduced from 0.873 to 0.031 at LD19, the absorbance of peaks at 1505~1515 cm^{-1} reduced from 0.875 to 0.026 at LD19, and the absorbance of peaks at 1455~1464 cm^{-1} reduced from 0.770 to 0.026 at LD19. The absorbance of peaks at 1379~1381 cm^{-1} reduced from 0.1.327 to 0.661 at LD20 extractives, the absorbance of peaks at 1365 cm^{-1} reduced from 0.599 to 0.036 at LD19 extractives, the absorbance of peaks at 1087~11174 cm^{-1} reduced from 1.399 to 0.060 at LD19 extractives, the absorbance of peaks at 853~886 cm^{-1} reduced from 1.202 to 0.030 at LD19 extractives. The results shown that the absorbance of each peak reached mostly at the benzene/ethanol extractives, minimally at the ethanol/methanol extractives.

Groups characteristics of three populus wood extractives of sequential extractions

Based on the above analysis on the extractives from single extraction, the three *populus* wood extractives of sequential extractions contained also O-H stretching in hydroxyl group, aromatic skeletal vibration plus C=O stretching, aromatic skeletal vibrations, aromatic skeletal vibration combined with C-H in plane deformation, syringyl vibrations, benzene ring-hydrogen bond vibrations.

To wood extractives of *Populus lasiocarpa*, the absorbance of peaks at 3355~3428 cm^{-1} increased from LD154, LD153, LD151, LD152, LD150, LD149, LD147, LD148 to LD147, but the stretching vibrations of the O-H were weaker. However, the stretching vibrations of the $-CH_2$ and $-CH_3$ were stronger. The absorbance of peaks at 1699 cm^{-1} reduced from LD147, LD148, LD149, LD150, LD151, LD152, LD153 to LD154, the absorbance of peaks at 1607 cm^{-1} reduced from LD147, LD148, LD149, LD150, LD152, LD151, LD153 to LD154, the absorbance of peaks at 1512 cm^{-1} reduced from LD147, LD148, LD149, LD150, LD152, LD151, LD153 to LD154, and the absorbance of peaks at 1462 cm^{-1}, 1383, 1271, 1177, and 823 cm^{-1} all reduced from LD147, LD148, LD149, LD150, LD151, LD152, LD153 to LD154. The results showed that the absorbance of peaks from LD147 extractives were the weakest, ones from LD154 extractives were the strongest.

To wood extractives of *Populus tomentosa*, the stretching vibrations of O-H, $-CH_2$ and $-CH_3$ were stronger, increased from LD171, LD176, LD175, LD174 to LD173, the absorbance of peaks at 1714 cm^{-1}, 1595, 1513, 1461, 1424, 1378, 1329, 1273, 1031, 853, and 772 cm^{-1} all reduced from LD173, LD174, LD175, LD176 to LD171, however, the absorbance of peaks at 1125 cm^{-1} reduced from LD174, LD173, LD175, LD176 to LD171. The results showed that the absorbance of peaks from LD173 extractives were the weakest, ones from LD171 extractives were the strongest.

To wood extractives of *Populus nigra*, the absorbance of peaks at 3341 cm^{-1} increased from LD189, LD187, LD186, LD188, LD185, LD183, LD184, LD182 to LD181, but the stretching vibrations of the O-H were stronger. The absorbance of peaks at 2928 cm^{-1} increased from LD188, LD189, LD187, LD186, LD185, LD184, LD183, LD182 to LD181. The absorbance of peaks at 1723 cm^{-1} increased from LD189, LD188, LD187, LD185, LD186, LD1184, LD1182, LD183 to LD181, the absorbance of peaks at 1597 cm^{-1} reduced from LD181, LD182, LD184, LD183, LD185, LD1186, LD188, LD187 to LD189, the absorbance of peaks at 1597 cm^{-1} reduced from LD181, LD182, LD184, LD183, LD185, LD1186, LD188, LD187 to LD189, the absorbance of peaks at 1459 cm^{-1}, 1421, 1276, 1125, and 1049 cm^{-1} all reduced from LD181, LD182, LD184, LD183, LD185, LD1186, LD188, LD187 to LD189. The results showed that the absorbance of peaks from LD181 extractives were the weakest, ones from LD189 extractives were the strongest.

CONCLUSIONS

Populus nigra wood, *Populus lasiocarpa* wood and *Populus tomentosa* wood were extracted and analyzed by FT-IR. And the groups characteristics of three populus wood extractives were found evidently. Among the single extractions, the three populus wood extractives contained

O-H, aromatic skeletal vibration plus C=O, aromatic skeletal vibrations, aromatic skeletal vibration combined with C-H in plane deformation, syringyl vibrations, benzene ring-hydrogen bond vibrations, the absorbance of each peak from wood extractives of *Populus nigra* reached mostly at the ethanol/methanol extractives, minimally at the benzene/ethanol extractives, the absorbance of each peak was different among the three wood extractives of *Populus lasiocarpa*, and the absorbance of each peak from wood extractives of *Populus tomentosa* reached mostly at the benzene/ethanol extractives, minimally at the ethanol/methanol extractives. Among the sequential extractions, to wood extractives of *Populus lasiocarpa*, the absorbance of peaks from LD147 extractives were the weakest, ones from LD154 extractives were the strongest; to wood extractives of *Populus tomentosa*, the absorbance of peaks from LD173 extractives were the weakest, ones from LD171 extractives were the strongest; to wood extractives of *Populus nigra*, the absorbance of peaks from LD181 extractives were the weakest, ones from LD189 extractives were the strongest.

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REFERENCES

1. Chunquan, Z., Jingpin, L., Xiaodong, L., Fuguo, W., Guizhen, C., 2001: The distribution and seasonal change of leaf area in poplar plantations managed in different ways. Scientia Silvae Sinicae 37(1): 46-51.
2. François, L., Agnès, L., Sven, de V., Jozef, T., 1998: Strategies for the conservation of a pioneer tree species, *Populus nigra* L., in Europe. Genet. Sel. Evol. 30(S1): S181-S196.
3. Lansheng, W., Wanxi, P., Minglong, Z., Zhi, L., 2013a: Separation characteristics of lignin from eucalyptus lignincellulose for medicinal biocellulose preparation. J. Pure Appl. Microbio 7: 59-66.
4. Lansheng, W., Wanx,i P., Zhi, L., Minglong, Z., 2013b: Molecule characteristics of eucalyptus hemicelluloses for medical microbiology. J. Pure Appl. Microbio 7(2): 1345-1349.
5. Lihua, S., Zhiqiang, Z., Chengfeng, L., Jinzhao, Z., Tonggang, Z., Xianrui, F., Jun, C., 2007: Sapflow of *P.* × *euramericana* cv plantation trees in Sandy Soil. Science of Soil and Water Conservation 5(1): 88-92.
6. Ningxia, D., Xin, L., Yun, L., Shouyi, C., Jinsong, Z., Da, H., Wenguang, D., Chunkui, S., Yingzhi, Z., Paula, M.P., 2012: Genetic transformation of *Populus tomentosa* to improve salt tolerance. Plant Cell Tiss. Organ. Cult 108: 181-189.
7. Qiu, X., Wanxi, P., Makoto, O., 2014: Molecular on ding characteristics of self-plasticized bamboo composites. Pak. J. Pharm. Sci. 27(4): 975-982.
8. Sheng-zuo, F., 2008: Silvi culture of poplar plantation in China: A review. J. Appl. Ecol. 19(10): 2308-2316.

9. Wanxi, P., Lansheng, W., Zhi, L., Minglong, Z., 2013: Identification and chemical bond characterization of wood extractives in three species of eucalyptus biomass. J. Pure Appl. Microbio. 7: 67-73.
10. Wanxi, P., Lansheng, W., Minglong, Z., Zhi, L., 2014a: Separation characteristics of lignin from *Eucalyptus camaldulensis* lignincelluloses for biomedical cellulose. Pak. J. Pharm. Sci 27(4): 723-728.
11. Wanxi, P., Qiu, X., Makoto, O., 2014b: Immune effects of extractives on bamboo biomass self-plasticization. Pak. J. Pharm. Sci 27(4): 991-999
12. Yong-Chang, S., Zhi, L., Wan-Xi, P., Tong-Qi, Y., Feng, X., Yi-Qiang, W., Jing, Y., Yang-Sheng, W., Run-Cang, S., 2014: Chemical changes of raw materials and manufactured binderless boards during hot pressing: Lignin isolation and characterization. BioResources 9(1): 1055-1071
13. Yoshinori, A., Eckhard, W., 1976: A novel phenolic acid derivative from buds of *Populus lasiocarpa*. Phytochem 15(5): 811-812.
14. Zsuffa, L., 1974: The genetics of *Populus nigra* L. Annales Forestales, Zagreb 6(2): 29-53.

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