INFLUENCE OF UREA FORMALDEHYDE RESIN ON THE PYROLYSIS CHARACTERISTICS AND GAS EVOLUTION OF WASTE MDF

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ABSTRACT

Pyrolysis behavior of medium density fiber board (MDF) with the presence of urea formaldehyde (UF) resin is different from the conventional biomass. To research the influence of UF resin on the pyrolysis of MDF, Thermo Gravimetric Analysis combined with Fourier Transform Infrared Spectrometry (TG-FTIR), Gas Chromatography coupled with Mass Spectrometry (GC-MS) and ultimate analysis were used to investigate pyrolysis process and products characteristics of MDF. As comparison, raw material wood fiber was also studied in this paper. It was showed that the process of MDF pyrolysis could be divided into three main phases. The degradation rate of MDF was higher than that of wood fiber, which might due to the influence of UF resin. The influence of UF resin during the pyrolysis process of MDF was mainly on the total release quantity of HNCO. The volume of releasing gas during pyrolysis process of MDF was larger than that of wood fiber. The nitric compounds in liquid products of MDF (amide, pyridine and pyrrole etc.) were more than those of wood fiber. Pyrolysis liquids were greatly influenced by the nitric compounds transferred from UF resin. Compared with wood fiber, MDF produced more nitric compounds in solid product which were mainly cyclic compounds, such as pyridine and pyrrole. As the nitric gasses mainly formed in the second stage, targeted pyrolysis conditions should be settled in this stage to decrease the formation of those gasses and make more N transfer into liquid or solid products to improve their application value.

KEYWORDS: Pyrolysis, MDF, UF resin, TG-FTIR, GC-MS.

INTRODUCTION

Pyrolysis is the thermochemical decomposition of organic materials at elevated temperatures without the participation of oxygen. There exists several techniques, in this paper, we focus on

the slow pyrolysis. Slow pyrolysis is the pyrolysis of biomass at slow heating rates (5–7 K.min-1). This method generates less liquid and gaseous products in favor of char production (Goyal et al. 2008). Pyrolysis is an effective and promising way for biomass decomposition, especially for wood materials. However, the pyrolysis process of waste wood-based board is not the same as conventional biomass materials due to the adhesive present in waste wood-based board, which influence the pyrolysis process and products (Girods et al. 2008a). Pyrolysis of wood starts at 200 and goes up to 500°C. The stage before 200 is called moisture evaporating stage, and the one after 500°C is calcination stage. Pyrolysis temperature of cellulose ranges from 300 to 375°C. On the other hand, hemicellulose whose weight loss occurs mainly between 225-325°C is less stable. The decomposition of lignin with complex structure takes place between 310-420°C (Liu 2005). Wu et al. (2010) has studied the pyrolysis process and the products of eucalyptus and corncob. Results show that cellulose, hemicellulose and lignin are the main chemical components of biomass, having different thermal degradation temperature ranges. Pyrolysis with proper conditions according to each main component characteristics is a reasonable and efficient way to use biomass.

Compared with wood, waste wood-based panel performs differently in pyrolysis. It may produce more nitrogenous products in pyrolysis because of the presence of adhesive. Some studies on pyrolysis behavior of resin show that the pyrolysis process of urea-formaldehyde resin residue could be subdivided into three stages: Drying the sample, fast thermal decomposition and further cracking process. 74 wt. % of the original mass loss occurs between 195 and 430°C in the second stage. The isocyanic acid emits at a low temperature is found as the most important nitrogencontaining gaseous product in pyrolysis, and there is a large amount of hydrocyanic acid emits at a higher temperature (Jiang et al. 2010). Pyrolysis of phenol formaldehyde resin has been investigated by Qian and Yu (2005). Several compounds, especially benzene, toluene, p-xylene could only be formed above 500-550°C. However, peak intensities for some phenol derivatives were decreased at a higher temperature.

Series of work have been done on pyrolysis of waste wood board by Girods et al. (2008a,b,c 2009a). In the experiments, temperature, air carrier, gas residence time were variable parameters. A two-step process of thermo-chemical conversion of wood board waste was discussed. The effect of the first step of the process (low-temperature pyrolysis which aims to remove nitrogen initially present in wood board waste) has been shown to be really efficient as the production of ammonia observed during the second step decreased slightly. The second step is pyrolysis and gasification of the pre-treated and virgin wood board sample between 800 and 1000°C to collect the liquid and gas products. The way of gasification has been shown to be more efficient in term of energy recovery but leads to a production of ammonia larger than in the case of the pyrolysis way. Concerning pre-treated samples, the best results have been obtained at 1000°C with samples pretreated at 250°C. Pyrolysis of waste wood board was reported by Mu et al. (2009). Research shows that wood vinegar is neutral to alkaline due to the influence of adhesives. Wood vinegar and charcoal from different raw materials have different characteristics in physical and chemical properties. Component analysis shows that there are more nitric compounds in pyrolysis liquid of wood board.

According to previous studies, the gas productions of biomass pyrolysis are mainly CO, CO_2 , H_2 , CH_4 and C_2H_4 . The gaseous products consist of both primary and secondary pyrolysis products. As the temperature increases, CO and CO_2 decreased, H_2 , CH_4 and C_2H_4 increased. Nitrogen compounds can be generated in biomass pyrolysis (Ren and Zhao 2009). Pyrolysis heating rates have influence on both nitrogen transformation rate and the transformation quantity. The higher the heating rate is, the lower the degree of nitrogen transformation will get. The main product of nitrogen transformation is NH_3 .

The liquid products of pyrolysis of wood and bamboo have extensive applications. Wood vinegar has been found to have the function of promoting plant growth, antibacterial, anticorrosion and also can be used for wood preservation (Wang et al. 2004). There are some differences between the pyrolysis condensate liquid of MDF and wood vinegar in composition and properties. The pyrolysis liquids of MDF are slightly alkaline because of the existence of nitrogen-containing compounds (Mu et al. 2011). The liquids can be used as basic solvents and additives in pesticides and in many other ways. A study on characteristic and bio-efficiency of the pyrolysis liquids from wood, particleboard, plywood and fiberboard shows that the inhibition of these liquids on the fungi are different due to different composition (Nakai et al. 2007).

The present work shows that particle board waste-derived activated carbons are efficient adsorbents for the removal of phenol from aqueous solutions with maximum measured capacities close to 500 mg.g⁻¹. Given the extremely low cost of activated carbons prepared from particle board waste, it should not be a problem to use it in higher amounts than commercial materials (Girods et al. 2009b).

Pyrolysis is believed to be a better utilization of waste wood-based board to convert it to energy rich products using suitable processes than other ways. How urea formaldehyde resin influences other components of MDF is an important issue. In this paper, TG-FTIR, ultimate analysis and GC-MS were mainly used to investigate the pyrolysis process, and products characteristics of MDF, and raw material wood fiber were also studied as comparison.

MATERIAL AND METHODS

Samples used in this experiment were MDF, wood fiber and UF resin with the molar ratio (F/U) of 1.08 provided by Beijing Wood Factory. Samples were crushed into the size lower than 0.2 mm and dried in the oven at 103°C for 3 h before the experiments.

TG-FTIR analysis

The experiment was carried out on Netzsch TG209 F1 thermo analyzer coupled with Nicolet 6700 FTIR at a linear heating rate of 20°C min⁻¹ from the ambient temperature to 800°C. The carrier gas was nitrogen with a flow rate of 60 mL. min⁻¹ in order to achieve an inert atmosphere. The tube and FTIR gas cell was maintained at 200°C in order to avoid any condensation of volatiles.

Ultimate analysis

To further explore the influence of UF resin in the pyrolysis of MDF, the samples of MDF, wood fiber and UF resin were pyrolyzed in the static bed with a temperature of 600°C for 2 h. Char was obtained for the later ultimate analysis.

The ultimate analysis of virgin samples, char of MDF, wood fiber and UF resin was carried on Elementar Vario EL III. Content of C, H and N was calculated by the mean value of two tests. Content of O was obtained by difference.

C	%				
Samples	С	Н	N	O*	
MDF	46.58	5.79	5.01	42.62	
Fiber	46.56	5.64	0.89	46.91	
UF	32.78	5.37	35.57	26.28	

Tab. 1: Elemental contents of MDF, wood fiber and UF resin.

Tab.2: Elemental contents of different chars.

	%			
Samples	С	Н	Ν	O*
MDF	78.64	2.11	1.18	18.07
Fiber	81.27	2.28	4.69	11.76
Fiber & UF	73.09	2.13	4.11	20.67
UF	61.79	2.08	20.17	15.96

GC-MS analysis

Liquid products were obtained from pyrolysis of MDF, wood fiber and UF resin by static bed with a heating rate of 5°C.min⁻¹ from the ambient temperature to 600°C, maintained for 30 min. The liquids were diluted in acetone to a concentration of 20 %.

Samples prepared from pyrolysis liquids were qualified by GC-MS. A column (RTX-5) with a diameter of 0.25 mm and length of 30 m was maintained at 60°C for 5 min, then the temperature was raised to 150°C with a heating rate of 12°C min⁻¹, after which temperature was raised to 260°C with a heating rate of 20°C.min⁻¹ and held at 280°C for 20 min. Nitrogen was used as carrier gas. The injection (injection volume of 1.0 μ l) was performed at 280°C in the split mode of (20:1). The mass spectrometer was operated in the electron ionization (EI) mode with ionization energy of 70 eV. Spectrum were analyzed by computer according to standard spectrum IST08.

RESULTS AND DISCUSSION

Analysis of pyrolysis process of MDF

As shown in Figs. 1 and 2, both the MDF and the wood pyrolysis processes can be divided into three stages. MDF weight loss rate was higher than that of wood fiber in the pyrolysis prophase while lower when entered the second phase. MDF had a smaller residual than wood fibers, this might be associated with UF resin in the pyrolysis process of MDF. Before heated to 320°C, the degradation rate of MDF is higher than wood fiber. This duration contributed to the most weight loss of both MDF and wood fiber.

MDF reaches its highest degradation rate of 15.98 % at the temperature of 360°C, while wood fiber reaches its highest degradation rate of 19.3 % at the temperature of 358°C. And the degradation rate of wood fiber is higher than that of MDF, but this only lasts for a short time (from 350 to 370°C), and then goes down. In this stage, the main degradation compounds is hemicelluloses and lignin. During pyrolysis, the group of amino and hydroxyl methyl crack. The carbonyl group also breaks in gas products such as CO and CO₂.

The total weight loss of 83 % of UF resin at 800°C is found in pyrolysis, among which 77 % is lost in the second stage between 220 and 400°C. The main degradation of UF resin takes

place in the second stage, from 220 to 400°C. Two peaks of this stage are found in spectrum: The first peak with the degradation rate of 11.5 % in 246°C, and the second peak with the highest degradation rate of 18.7 % in 288°C, which agrees with literature (Jiang et al. 2010).

The degradation rate of MDF performs in a middle level between UF resin and wood fiber, which means both UF resin and wood fiber may influence the MDF pyrolysis. In the whole degradation, MDF weight loss rate is higher than wood fiber. It is found from Fig. 1 that the degradation rate of UF resin is higher than wood fiber, which might be a reason for the promoting role of UF resin in MDF pyrolysis. And the gas products might form a different reacting environment so that influenced the MDF pyrolysis process.

MDF weight loss rate is higher than wood fiber but lower than wood (Feng et al. 2012). We may conclude that UF resin promotes the pyrolysis of cellulose, hemicelluloses and lignin but prevents the compound which removes in the fiber production procedure from wood. This speculation need to be confirmed by further research.

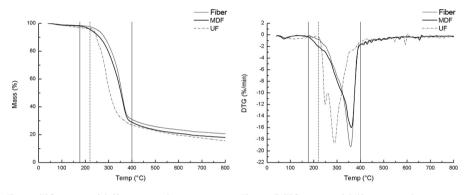


Fig. 1: TG curves of different samples. Fig. 2: DTG curves of different samples.

The N transformation to pyrolysis products of MDF

Waste MDF with UF resin in it releases more nitric gasses in pyrolysis. Finding out the releasing process and characteristic of nitric gasses can help to decrease the formation of those gasses, and make more N transfer into liquid or solid products to improve their application value.

N transformation into composition of pyrolysis gasses

The characteristic frequencies used for the qualitative and quantitative analysis were 2300 to 2400.cm⁻¹ for carbon dioxide(CO₂), 2000 to 2250.cm⁻¹ for carbon monoxide (CO), 800 to 1200.cm⁻¹ for ammonia (NH₃), and 2200 to 2300.cm⁻¹ for isocyanic acid (HNCO). Gas analysis was also done by matching the spectra against those from the spectrum library Nicolet TGA Vapor Phase of the software Ominic combined with the methods reported by literature (Girods et al. 2008).

The gaseous products of different samples during thermal process were detected on-line by TG-FTIR. The typical 3D infrared spectra of volatile products from pyrolysis of MDF, wood fiber and UF resin are shown in Fig. 3. In general, there are more kinds and longer duration of pyrolysis gas generating from MDF than wood fiber and UF resin, especially the CO_2 and HNCO. As a result, the total gas quantity of MDF is more than others.

Gas releasing temperature of wood fiber ranges from 320 to 440° C. There are a few kinds of pyrolysis gasses from UF resin and the releasing temperature ranges mainly from 240 to 400° C.

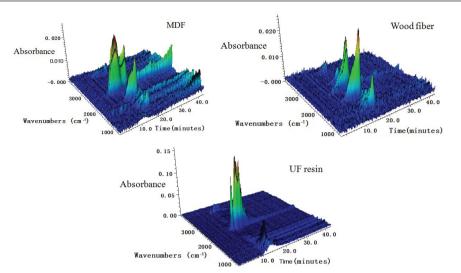
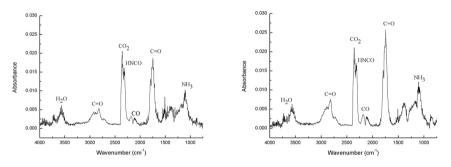


Fig. 3: Typical 3D infrared spectra of volatile products from pyrolysis of volatile products from pyrolysis of different materials.



highest intensity value for MDF.

Fig. 4: Spectrum of gasses released out at the Fig. 5: Spectrum of gasses released out at the highest intensity value for wood fiber.

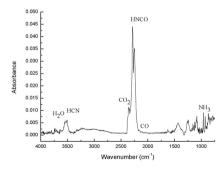


Fig. 6: Spectrum of gasses released out at the highest intensity value for UF resin.

Figs.4-6 are the representative of FTIR spectrum of the three samples in pyrolysis temperature of 360°C when MDF releases gas reached its highest intensity value. Main gaseous products and functional groups can be determined through the characteristic peaks in the spectra. Characteristic peaks related to main gasses of CO, CO_2 , HNCO and NH₃ can be easily distinguished in all FTIR spectra. The absorbance peaks in wood at 1700-1800 and 2750-3000.cm⁻¹ are most likely responsible for the carboxyl and carbonyl comparing with the spectrum library. Absorbance peak at 3450-3600.cm⁻¹ in the spectrum of UF resin represent the release of HCN. There are fewer kinds of gas composition in UF resin pyrolysis gasses.

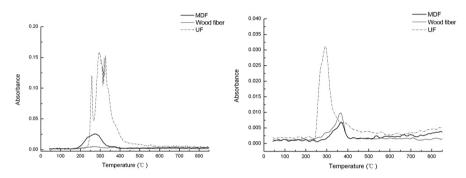


Fig. 7: Evolution of HNCO with temperature Fig. 8: Evolution of NH_3 with temperature during the pyrolysis. during the pyrolysis.

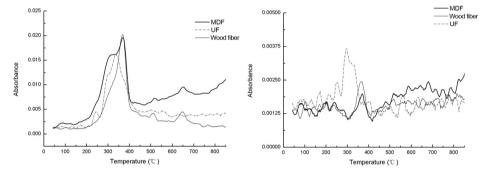


Fig. 9: Evolution of CO_2 with temperature Fig. 10: Evolution of CO_2 with temperature during the pyrolysis. during the pyrolysis.

Figs. 7-10 shows the releasing process of HNCO, NH_3 , CO and CO₂ in three samples during pyrolysis. It can be found that the degradation of urea function in UF resin generates HNCO and the precursor of HCN, which crack into HCN under 650°C (Qian and Yu 2005). HCN might be therefore much difficult to be detected over the pyrolysis process while the HNCO peak was obvious. The HNCO intensity of UF resin is much higher than MDF and wood fiber. The first peak appears at 250°C while the double-peak with the highest degradation rate appears between 300 and 350°C. The groups of amino and hydroxyl methyl crack during pyrolysis, then bond with free hydrogen to form HNCO (Feng et al. 2012). The HNCO intensity of MDF is higher than that of wood fiber, and it may come from UF resin as its forming temperature range is similar with that in UF resin.

 $\rm NH_3$ can be found in all of the three figures. The element analysis suggests that there are N compound in wood fiber, which could be another origin for $\rm NH_3$.

From the absorbance curves of wood fiber and MDF, CO_2 and small amount of CO can be seen. But from the absorbance curves of UF resin we can only see very little CO_2 . The CO_2 releasing rate of MDF is quite high with a large volume. This may be influenced by both UF resin and wood fiber. CO_2 peak appears at 360°C, in this stage the degradation of cellulose and lignin mainly take place, CO_2 may come from the carboxyl of them. And CO_2 forms during the pyrolysis of UF resin (Jiang et al. 2010). The carbonyl in UF resin may also break and form CO_2 and CO.

The influence of UF resin during the pyrolysis process of MDF acts mainly on HNCO. But the influence on NH_3 is weak. The HNCO volume is far more than NH_3 , so the difference of NH_3 between MDF and fiber is not obvious. The maximum intensities of HNCO and NH_3 in FTIR spectrum agrees well with the maximum weight loss rate of UF resin DTG profile. HNCO and NH_3 only form in the second stage. But CO and CO₂ releases a little mount in the third stage which may produce in the degradation of lignin and former pyrolysis products. In further research, various factors in the second stage should be studied to control the N products.

N transformation into composition of pyrolysis liquid

Main compositions of different pyrolysis liquids analyzed by GC-MS are listed in Tab. 3. The GC-MS study indicates that pyrolysis liquids of MDF contain more nitric compounds than that of wood fiber which is rich in acids, alcohols, phenols and ketones. MDF pyrolysis liquids contain four kinds of compounds, containing N in the form of amide and eight compounds containing N in ring form such as pyridine and pyrazine, some of which are ketones. This may illustrate the impact of UF resin material on ketones. There is another compound of N with a benzene ring which may come from benzene propane which is the main structure units of lignin. These linear and cyclic nitric compounds might be generated from decomposition and reaction in the pyrolysis process of UF resin added in the wood-based panel. Pyrolysis liquids of wood fiber have a few nitric compounds which are mainly amides of small molecules. Except for some phenols, alcohols and ketones, the rest compounds of UF resin pyrolysis liquids are all nitric compounds. Contents of N, N-dimethyl urea and urea in UF resin pyrolysis liquid are high, which may come from the formaldehyde and urea in UF resin. And there are also two kinds of amides and 7 kinds of pyridine ring structures.

The pyrolysis process of wood is very complicated, including the thermal degradation of cellulose, hemicellulose and lignin. Wood vinegar contains 16 kinds of ketones, 14 kinds of phenolic and other compounds like esters, aldehydes and alcohols besides the main components acetic acid (Xu et al. 2006). In addition, there are nitric components in wood vinegar like amine, methylamines, dimethylamine and pyridine of trace content (Wang et al. 2004). The result of this paper is consistent with the mentioned studies and shows that pyrolysis liquids are influenced a lot by the nitric compounds transferred from UF resin.

The distinctive nitric compounds from MDF reveals the chemical interaction between UF resin and wood components. Such nitric compounds make pyrolysis liquids of waste wood-based composites different from those of general biomass and influence the utilization of pyrolysis liquids.

Some compounds generated in MDF producing heat pressing degrade into products that are different from pyrolysis liquids of wood fiber. Besides, due to the presence of UF resin, it might form an alkaline pyrolysis environment that is different from wood fiber, which can also influence the pyrolysis products. And the prime products of UF resin react with wood fiber and transform new products.

Groups	MDF	Wood fiber	UF resin
Acids	Acetic acid	Acetic acid	-
	Propanoic acid	Propanoic acid	-
	-	Butanoic acid	-
Esters	Butyrolactone	Butyrolactone	-
	deltaValerolactone	Butanoic acid, 2-propenyl ester	-
Alcohols	Furfurylalcohol, tetrahydro-	Furfurylalcohol, tetrahydro-	2-Nitro-2-methylpropanol
	-	1-Cyclopropyl-1-pentanol	-
Phenols	Phenol	Phenol	Phenol
	Phenol, 2-methoxy-	Phenol, 2-methoxy-	Phenol, 2-methoxy-
	Phenol, 2,6-dimethoxy	Phenol, 2,6-dimethoxy-	Phenol, 2-methoxy-4-methyl-
	-	-	Phenol, 4-ethyl-2-methoxy-
	-	-	Phenol, 3-methyl-6-propyl
	2-Cyclopenten-1-one,	2-Cyclopenten-1-one,	2-Cyclopenten-1-one,
Ketones	3-methyl	2-methyl	2,3-dimethyl
	-	Ethanone, 1-(2-furanyl)	-
		2-Cyclopenten-1-one,	
	-	3-methyl	-
		2-Furanone, 2,5-dihydro-3,5-	
	-	dimethyl	-
		2-Cyclopenten-1-one,	
	-	2,3-dimethyl	-
N-Compounds	Acetamide	Acetamide	Pyrrole, 1-methyl
iv compounds	Pyridine, 2-methyl	Acetamide, N-methyl	Pyrrole
	Acetamide, N-methyl	Acetamide, N,N-dimethyl	Formamide, N,N-dimethyl
	Succinimide, N-methyl	Succinimide, N-methyl	Pyridine, 2-methyl
	2H-Azepin-2-one, hexahydro-	Succiminac, it methyr	Pyrazine, methyl
	1-methyl	N-Methylcaprolactam	
	3-Hydroxypyridine	3-Hydroxypyridine	Acetamide, N-methyl
	Phenol, 3-amino	2-Methyl-3-hydroxypyridine	Pyrimidine, 4-methyl
	Piperidine, 1,2-dimethyl	2 memyr-3-nyuroxypyriume	Pyridine, 2-ethyl
	1-Azabicyclo[2.2.2]octan-4-ol		Propanenitrile, 2,2-dimethyl
	Hydrouracil, 1-methyl	-	Pyridine, 2,5-dimethyl
	Phenol, 2,6-dimethoxy	-	Benzenamine, N,N-dimethyl
	1,3,5-Triazine-	_	1,3,5-Triazine-
	2,4,6(1H,3H,5H)-trione,	-	2,4,6(1H,3H,5H)-trione,
	1,3,5-trimethyl		1,3,5-trimethyl
	-	-	2(1H)-Pyridinone, 1-methyl
	-	-	Urea, N,N-dimethyl
	-	-	Urea, formyltrimethyl
	-	-	Urea, N,N'-dimethyl
	-	-	Urea, methyl

Tab. 3: Main components of different pyrolysis liquids.

N transformation into the composition of pyrolysis char

According to our previous work, the results of products yield and ultimate analysis show that the N content of MDF is 5.01 %. 2/3 of nitric compounds decompose or form during pyrolysis process and transform into gas or liquid products. As a result, N content in the solid residue

residual i 1.77 %. Wood fibers has a lower N content which is 0.89 % and the content goes down to 0.43 % after the pyrolysis while the remained N transforms into gas or liquid products. N content of UF resin is 35.57 % and it decreases to 2.10 % after the pyrolysis when most of the N transformes into gas or liquid products. The existing forms of nitrogen compounds in MDF are mainly structures of primary amines and amide, with concentrations of 4.28 and 1.07 % respectively. The existing forms of nitrogen compounds in MDF char are pyridine and pyrrole, with concentrations of 6.65 and 7.45 % respectively.

CONCLUSIONS

The results showed that MDF pyrolysis process could be divided into three main phases. The degradation rate of MDF was higher than that of wood fiber, which mainly due to the influence of UF resin.

Gas composition of MDF and wood fiber are similar, but the volume of gas products of MDF is more than that of wood fiber. The influence of UF resin during the pyrolysis process of MDF is mainly on the total release quantity of HNCO. Being with or without UF resin makes not that much difference in the formation of NH_3 . The releasing trend of CO and CO₂ in MDF and wood fiber is almost same, just having little difference in the amount. As the nitric be gases mainly formed in the second stage, targeted pyrolysis conditions should be settled in this stage to decrease the formation of N-contained be gases and make more N transfer into liquid or solid products to improve their application value.

There are more nitric compounds in solid products and liquid products of MDF than wood fiber, which are mainly cyclic compounds such as pyridine and pyrrole. Pyrolysis liquids of MDF were influenced a lot by the nitric compounds transferred from UF resin.

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