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ORIGINAL ARTICLE

The water resistance of corrugated paper improved by lipophilic extractives and lignin in APMP effluent

Liying Dong[1] · Huiren Hu[1] · Fei Cheng[1] · Shuo Yang[1]

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Abstract In this article, quantitative determination of compositions (hemicellulose and lignin) in the APMP (alkaline peroxide mechanical pulping) effluent was done. The MTBE (methyl tert-butyl ether) extracts of the APMP effluent (extractives) were analyzed by GC/MS (gas chromatography–mass spectrometry). The result of GC/MS showed that fatty acids (C14–C28) and their esters, sterols were the main components of extractives. Based on the qualitative and quantitative determination of compositions in APMP effluent and the mechanism of surface sizing, APMP effluent combined with starch and $Al_2(SO_4)_3$ was used as corrugated paper surface sizing agent; the water resistant mechanism of which was explored. Combined effects of lignin (extractives) and Al^{3+} played a major role in hydrophobic properties of corrugated paper, the Cobb values of corrugated paper sized by which were 35.2 and 39.8 g/m^2, respectively. The water contact angles were 126.6° and 115.6° at 120 s, respectively. No previous studies or article on APMP effluent improving the water resistance of corrugated paper have been reported.

Keywords Alkaline peroxide mechanical pulping effluent (APMP effluent) · Corrugated paper · Surface sizing agent · Water resistance

✉ Liying Dong
dl_dongliying@126.com

1 Tianjin Key Laboratory of Pulp and Paper, Tianjin University of Science and Technology, No. 29, The 13th Avenue, Tianjin Economic and Technological Development Area, 300457 Tianjin, People's Republic of China

Introduction

In recent years, many new APMP and P-RC APMP lines (preconditioning followed by refiner chemical treatment alkaline peroxide mechanical pulping lines) have been installed in China, and a large amount of APMP effluent has been produced. Although the yield of pulp is 90 %, which still has about 10 % organic and inorganic matters dissolved in the effluent and a small amounts of them is adsorbed on the pulp [1]. In previous studies, researchers have paid more attention to their negative impact on the pulp and paper mill process, which may cause production and environmental problems in the pulp and paper industry. In the pulping process, these organic matters, especially wood extractives and lignin degradation products, are difficult to remove in the washing stages and may lead to sticky deposits on process equipment. The accumulation of small amounts of these substances can result in blockages, which are responsible for reduced production levels, higher equipment maintenance costs, higher operational cost, and increased incidence of quality defects [2]. Once, these extractives or lignin enter into wet end together with the pulp, most of which are in the form of colloidal and dissolved particles [3]. Only fatty and resin acids and sterols are found in dissolved form in these waters [4]. In colloidal resin particles, steryl esters and triglycerides, which are the most hydrophobic components of the extractives, form the hydrophobic core while fatty and resin acids and sterols form the thin surface layer of the particles. The carboxyl groups of fatty and resin acids are orientated toward the aqueous phase [5–8]. The particles are negatively charged in the pH 2–11. Carboxyl and hydroxyl groups on the surface of colloidal resin particles stabilized them electrostatically, while hemicelluloses especially mannans stabilize them sterically. In wet end, with a high degree of white-water system closure, these organic matters can have a negative impact on paper machine runnability and product quality. Problems and distances such as pitch depositions on the paper, specks in the paper, decreased wet strength, interference with cationic process chemicals, and impaired sheet brightness and paper strength are often caused.

Although these substances in APMP effluent have so many disadvantages, we hope to explore their potential usefulness in the papermaking process, which requires a thorough analysis of all components in the APMP effluent. The knowledge of the chemical nature of these organic components will assist the development of suitable methods for their utilization.

In this work, quantitative determination of compositions (hemicellulose and lignin) in the APMP effluent was done. The extracts of the APMP effluent [methyl tert-butyl ether (MTBE) extractives] were qualitative and quantitative analysis by GC/MS (gas chromatography-mass spectrometry). Based on the qualitative and quantitative determination of compositions in the APMP effluent and the mechanism of surface sizing, APMP effluent combined with starch and $Al_2(SO_4)_3$ was used as corrugated paper surface sizing agent, the water resistant mechanism of which was explored. No previous studies or articles on APMP effluent improving the water resistance of corrugated paper have been reported.

Materials and methods

Materials

The APMP effluent in this study (pH = 7.12, concentration 14.5 %) was poplar APMP pulping effluent supplied by a paper mill in the Shandong province in China. The corrugated paper and starch were supplied by a paper group in Tianjin, China. The other chemicals used in this study were analytical reagents obtained from Tianjin Kermel Chemical Reagent Development Center in China.

Methods

Qualitative and quantitative determination of compositions in the APMP effluent

The solid content of APMP effluent was determined according to TAPPI standard. The contents of hemicellulose and lignin were determined by gravimetric method. The qualitative analysis of hemicellulose and lignin was determined by FT-IR (Fourier Transform Infrared Spectroscopy). The FT-IR spectra were recorded with a FTIR-650 scanning from 4000 to 400 cm^{-1} with a resolution of 2 cm^{-1} using KBr pellets at room temperature. Extractives were separated according to the previously reported literature [6]. The identification of the extractives was performed using GS/MS. The scheme for analysis of compositions of the APMP effluent was outlined in Fig. 1.

Extractives derivatization and GC/MS conditions

For the identification of wood extractives in APMP effluent by GC/MS, a derivatization step was necessary. APMP effluent was diluted with distilled water to a TOC (total organic carbon) concentration of about 200 mg/L. 4 mL APMP effluent was added 0.05 M sulphuric acid to adjust the pH to about 3.5. Then 2.0 mL MTBE was added. The sample was vigorously shaken by hand for 1 min and was then centrifuged at 4000 rpm for 5 min. The clear MTBE layer was carefully pipetted off. In some samples, a thin emulsion layer remained at the phase boundary. This layer was left in the test tube. The extraction layer was repeated twice with 2 mL portions of pure MTBE. The combined MTBE solution was evaporated in a stream of nitrogen. The residues were added 80 μL BSTFA (bis(trimethylsilyl)-trifluoro-acetanide) and 40 μL TMCS (trimethylchlorosilane). The solution was kept in an oven at 70 °C for 20 min and was thereafter ready for analysis by GC/MS [9].

The GC/MS analysis of the extractives was performed on a varian MS4000, VF-5 ms (30 m), the oven was heated from 100 to 270 °C at 5 °C/min and then heated from 270 to 300 °C at 10 °C/min held for 5 min. The transfer line was kept at 300 °C. Helium was used as carrier gas. The compounds were identified by comparing the mass spectra thus obtained with those of wiley and NIST05 computer libraries, by mass fragmentography.

Surface sizing and the water resistance of corrugated paper

The surface sizing agent (starch:APMP effluent: $Al_2(SO_4)_3$ = 2:2:1 based on solid content) was used to size the corrugated paper (100 g/m^2). The concentration of sizing agent was 8 %. Starch viscosity was 66 cp. Other sizing agents viscosity 30–40 cp. The sizing was performed using a laboratory coater on the surface of the corrugated paper with a size press pickup of 6 g/m^2 at the temperature of 65 °C. The sized paper sheets were dried with a dryer at 118–127 °C. The water resistance was measured using a Cobb tester, which was used to represent the amount of water absorbed by the paper after bearing water with a weight of 100 g for 60 s at room temperature.

Dynamic contact angle analysis

Dynamic contact angle (DCA) measurements were performed within the timeframe (30 s for original paper and

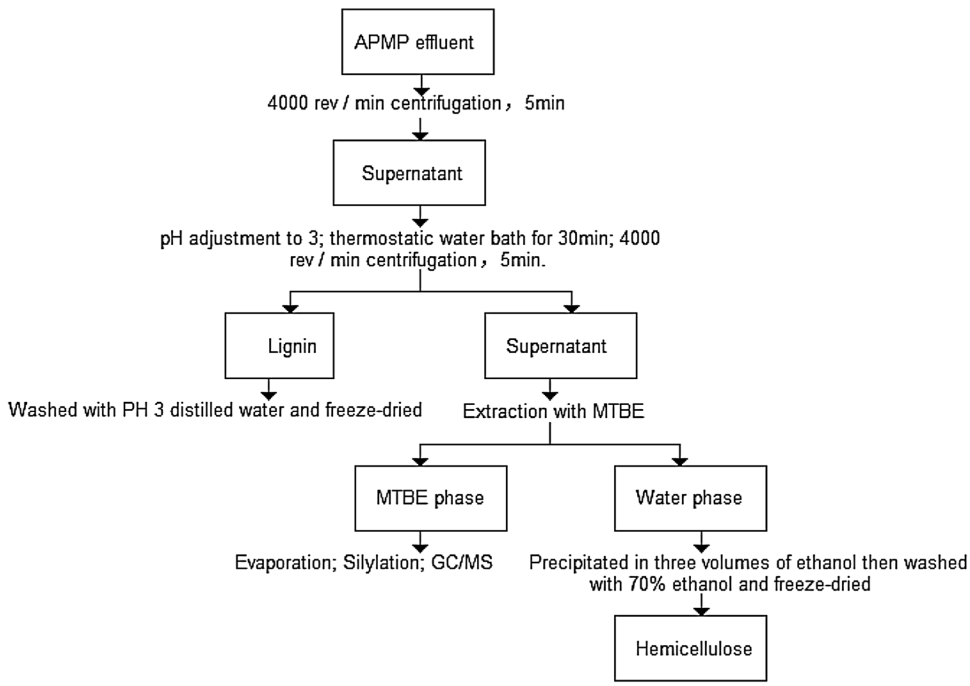

Fig. 1 Scheme for analysis of compositions in the APMP effluent. *APMP* alkaline peroxide mechanical pulping, *MTBE* methyl tert-butyl ether, *GC/MS* gas chromatography–mass spectrometry

120 s for sized paper). The liquid used in contact angle measurements was distilled water, and measurements were taken at 5 s (original paper) and 20 s (sized paper) intervals from the time the water droplet first made contact with the paper substrate.

Results and discussion

Qualitative and quantitative determination of compositions in the APMP effluent

In the process of APMP pulping, many substances are dissolved, such as carbohydrates, lignin, as well as lipophilic extractives. To utilize APMP effluent as a feedstock for production of higher value added products, it is essential for the qualitative and quantitative analysis of compositions in the APMP effluent (Table 1).

The results showed the solids content of APMP effluent was 14.5 %; 6.75 % (47 % based on the solid content of APMP effluent) and 2.92 % (20 % based on the solids content of APMP effluent) of hemicellulose and lignin were isolated from APMP effluent, respectively. The qualitative analysis of hemicellulose and lignin was determined by FT-IR spectra. The FT-IR spectra results were presented in Fig. 2. The ash content of APMP effluent was 31 %. The others were lipophilic extractives. The chemical compounds identified of extractives were by GC/MS. The GC/MS analysis results of extractives were presented in Fig. 3 and Tables 2, 3.

Table 1 The content of main compositions in APMP effluent

	Solids	Hemicellulose	Lignin	Extractives	Inorganics
Content (%)	14.5	6.8	2.9	2.4	2.4
Relative content (%)	100	46.9	20.1	16.5	16.5

In Fig. 2a, FT-IR results showed the original absorptions at 3415, 2829, 1637, 1405, 1081, 1052, 860, 620 and 545 cm^{-1} were typical of hemicellulose [10, 11], which can fully explain the ethanol precipitation of APMP effluent are mainly xylans. In Fig. 2b, a strong hydrogen band (–OH) stretching at 3430.5 cm^{-1} and C–H stretching at 2927.4, 2850.3 cm^{-1}. The absorption band at 1718.3 cm^{-1} represents the stretching of C=O including carboxyls, carbonyls and quinones in lignin. The vibration of aromatic ring is assigned to 1596.8, 1508.1, 1419.4 cm^{-1}, which is around 1600–1400 cm^{-1}. The bands at 1267.0 cm^{-1} are corresponding to guaiacyl units of lignin, while the bands at 1224.6, 1124.3 cm^{-1} attribute to syringyl units of lignin [12, 13]. The FTIR spectrum indicates that the main ingredients of acid precipitate in APMP effluent are lignin.

GC/MS results (Fig. 3) showed that APMP effluent contained a large number of extractives. Thirty compounds had been identified and classified in the single chromatogram. Meanwhile extractives also contained comparable lignans and low-molecular-weight hydroxy acids.

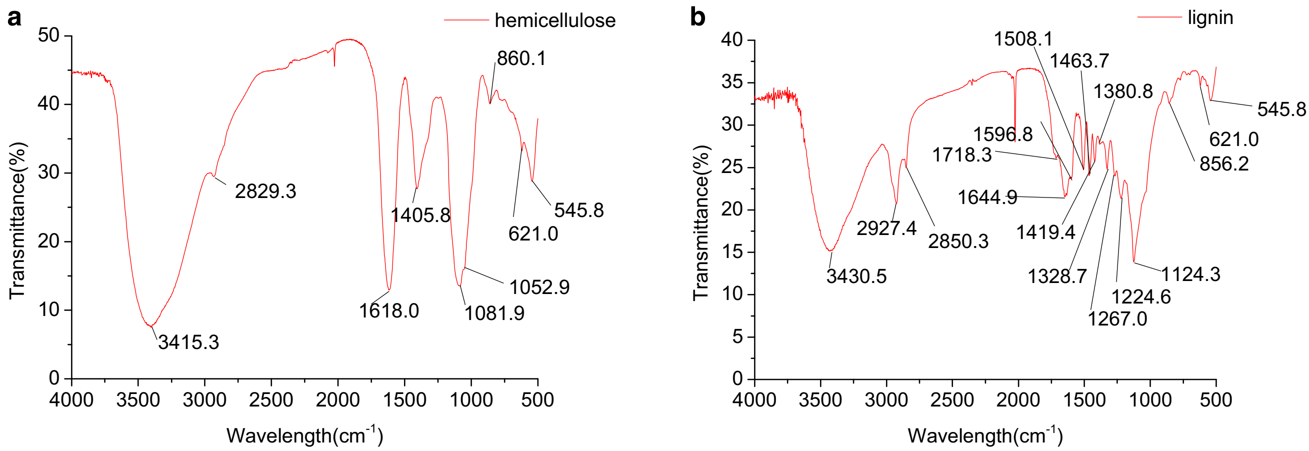

Fig. 2 FT-IR spectra of hemicellulose and lignin

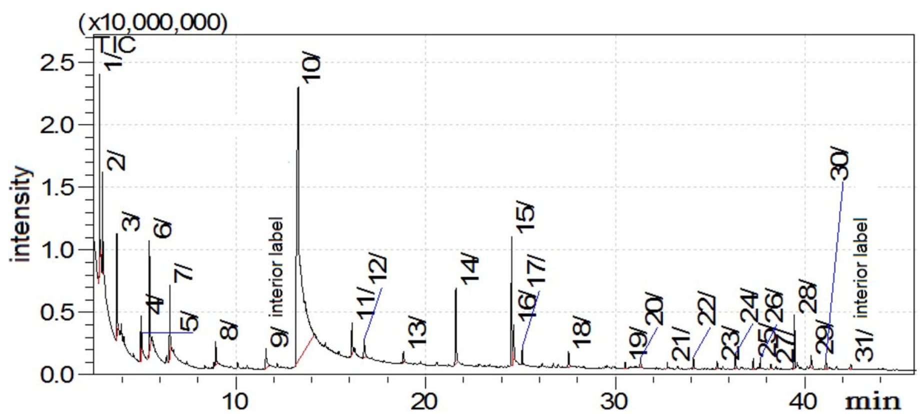

Fig. 3 Chromatogram of MTBE extractives

Extraction with MTBE, both phenolic low-molecular-mass components and lipophilic were extracted in high yield by MTBE [9]. These latter components were classified into resin acids, fatty acids, sterols, glycerides. Fatty acids were shown to be the main group of lipophilic extractives followed by sterols. The amounts of fatty acids and sterols were relatively high in wood plants because most of these compounds exist in esterified form. Sterol esters are formed by sterols and fatty acids. The common saturated fatty acids (undecanoic acid (C11:0), palmitic acid (C16:0), stearic acid (C18:0), behenic acid (C22:0), lignoceric acid (C24:0), cerotic acid (C26:0), octacosanoic acid (C28:0)) and the unsaturated fatty acids (9,12-octadecadienoic acid (C18:2) and linoleic acid (C18:3)) were found in APMP effluent. Sterols were found to be the second main group dominated by stigmastanol. Others were beta-Amyrin and cyclolaudenol in lesser amouts.

Theory of sizing

Sizing is a widely used process to impart water resistance to paper and paperboard by treating fiber substrate with hydrophobic substance. There are two types of sizing: surface sizing and internal sizing. Internal sizing chemicals used in papermaking at the wet end are Alkyl ketene dimer (AKD) [14–16], alkyl succinic anhydride (ASA) [17] and rosin [18]. Surface sizing agents consist of mainly modified starches and other chemicals, such as waterborne polyurethane [19, 20], styrene/acrylic type polymers [21–23]. Surface sizing agents are amphiphilic molecules having both hydrophilic (water-loving) and hydrophobic (water-repelling) ends. The sizing agent adheres to fibers substrate and forms a film, with the hydrophilic tail facing the fiber and the hydrophobic tail facing outwards, resulting in a smooth finish that tends to be water-repellent, which can prevent water to wetting the paper sheet. What is wetting? Wetting is the ability of liquids to form interfaces with solid surfaces. To determine the degree of wetting, the water contact angle that is formed between the liquid and the solid surface is measured, which is a good indicator of wetting or dewetting. The smaller the water contact angle and the smaller the surface tension, the greater the degree of wetting [24]. The Young's equation describes wetting if $0° \leq \theta < 90°$ and non-wetting if $\theta > 90°$, which mean a

Table 2 Main compositions and concentrations of MTBE extractives

Peak no.	Retention time (min)	Compound	Concentration (mg/mL)
1	2.812	Lactic acid	1.189
2	2.974	Glycolic acid	0.722
3	3.731	3-Hydroxypropionic acid	0.657
4	4.989	2-Hydroxyisocaproic acid	0.320
5	5.046	2-Hydroxy-3-methylpentanoic acid	0.138
6	5.440	Benzoic acid	0.860
7	6.513	Succinic acid	0.477
8	8.927	Salicyl alcohol	0.224
9 (interior label)	11.581	Undecanoic acid	0.277
10	13.274	P-hydroxybenzoic acid	12.231
11	16.113	Vanillic acid	0.293
12	16.767	Azelaic acid	0.204
13	18.842	Syringic acid	0.098
14	21.595	Palmitic acid	0.730
15	24.545	Linoleic acid	1.253
16	24.651	Linoleic acid	0.410
17	25.094	Stearic acid	0.153
18	27.541	Dehydroabietic acid	0.173
19	30.529	Hexadecanoic acid glyceride	0.130
20	31.331	Behenic acid	0.194
21	32.749	Lignoceric acid	0.105
22	34.121	Lignoceric acid	0.256
23	35.368	Cerotic acid	0.169
24	36.342	Cerotic acid	0.306
25	37.276	Octacosanoic acid	0.165
26	37.640	Glycerol	0.165
27	38.206	Lignoceric acid	0.104
28	39.446	Stigmasterol	1.243
29	40.334	Beta-amyrin	0.361
30	41.108	Cyclolaudenol	0.322
31 (interior label)	42.417	Lupa-13(18),22-dien-3-ol,acetate	0.167

Table 3 Main compositions and relative amount of lipophilic extractives

Main compound type of lipophilic extractives	Concentration (mg/mL)	Relative content (%)
Resin acids	0.173	2.7
Fatty acids	3.845	63.7
Sterols	1.926	31.6
Glycerides	0.13	2.0
Total content	23.65	100

water drop spreading out to increase the contact surface on a hydrophilic surface, but minimizing the contact surface on a hydrophobic surface. By the same token, for paper or paperboard, the greater the water contact angle, the better the water resistance. From the Young's equation, the high surface tension of solid is more easily wetted than that of low surface tension. Surface sizing agents are amphiphilic molecules, having both hydrophilic (water-loving) and hydrophobic (water-repelling) ends. The sizing agent adheres to substrate fibers and forms a film, with the hydrophilic tail facing the fiber and the hydrophobic tail facing outwards, resulting in a smooth finish that tends to reduce the surface tension of the sheet, which make the paper sheet water-repellent and which is also the surface sizing mechanism.

The results of APMP effluent composition analysis show that the content of lignin is 2.9 % (20.1 % based on the solid content of APMP effluent). As everyone knows, lignin contains hydrophobic phenylpropane structure and hydrophilic hydroxyl group. The main components of extractives in APMP effluent are fatty acids and sterols, which are all C16–C28 amphiphilic molecular. The hydrophilic groups are hydroxy and carboxy (–COO–). The following research was done. The paper was sized with sizing agent (starch:APMP effluent:$Al_2(SO_4)_3$ = 2:2:1 based on solid content). Whether we can get our desired results: the polar hydrophilic groups are combined with the paper fibers by Al^{3+}. At the same time, the hydrophobic phenylpropane and C16–C28 can stretch orient to face outwards and form a continuous film layer, which tends to water-repellent.

Is the result we expected? The effect of APMP effluent on corrugated paper physical strength properties has been studied more by our research team in the past researches. Here we list them, but not discussed in detail. We focus on the influence of APMP effluent on the water resistance of corrugated paper, which assesses by Cobb value and dynamic contact angle.

The water resistance of corrugated paper

The Cobb value of corrugated paper

The sizing agent (starch: APMP effluent: $Al_2(SO_4)_3$ = 2:2:1) was used to size the corrugated paper. The Cobb value of sized corrugated paper was reduced to 22 g/m^2 compared with that of original corrugated paper 127 g/m^2. However, the corrugated papers were sized by the sizing agent of APMP effluent free (starch: $Al_2(SO_4)_3$ = 4:1 and 100 % starch), which showed high Cobb value of 102 and 124 g/m^2. The above experimental phenomena illustrated in Table 4 that certain substances in APMP effluent can improve the water resistance of corrugated paper.

To prove our conjecture, the components of APMP effluent were separated. Figure 1 shows the separation process. Effect of various components (hemicellulose, lignin and extractives) on the water resistance of the corrugated paper was assessed. The proportion of various components in the sizing agent was consistent with the sizing agent (starch: APMP effluent: $Al_2(SO_4)_3$ = 2:2:1). Table 5 shows both lignin and extractives have contribution to the water resistance of corrugated paper. The Cobb values of corrugated paper sized by the sizing agents (72 % Starch +8 % Lignin + 20 % $Al_2(SO_4)_3$ and 78.8 % Starch +1.2 % Extractives + 20 % $Al_2(SO_4)_3$) were reduced to 35.2 and 39.8 g/m^2, respectively.

Dynamic contact angle measurement

Corrugated papers sized separately by lignin or extractives combined with starch and $Al_2(SO_4)_3$ had low Cobb value, which showed that both of them contribute to the water resistance of corrugated papers. So it is necessary to assess separately the effect of extractives and lignin on the surface energy of the corrugated paper via DCA analysis. Due to the extractives or lignin deposition onto the cellulose fibers, the actual resultant changes to the surface energy of the corrugated paper substrate are largely unknown. Therefore, the behavior of water droplets after contacting with the sized papers is just as important as the water contact angle made with the surface. Contact angle can indicate a decreased degree of surface tension of paper and further indicate the water resistance degree of corrugated paper. Small contact angles ($\ll 90°$) correspond to poor water resistance, while large contact angles ($\gg 90°$) correspond to good water resistance [25].

Table 4 Effect of APMP effluent on the physical properties and the water resistance of corrugated paper

	Sizing pickup (g/m^2)	Tensile index (N m g^{-1})	Ring crush index (N m g^{-1})	Burst index (kPa m^2 g^{-1})	Cobb value (g m^{-2})
1		34.8	3.5	1.0	127
2	5.3	42.8	5.9	1.4	102
3	5.8	49.2	5.5	1.5	22
4	5.8	50.0	5.4	1.4	124

1 Original paper, *2* 80 % Starch + 20 % $Al_2(SO_4)_3$, *3* 40 % Starch + 40 % APMP effluent + 20 % $Al_2(SO_4)_3$, *4* 100 % starch

Table 5 Effect of various components in APMP effluent on the physical properties and the water resistance of corrugated paper

	Sizing pickup (g/m^2)	Tensile index (N m g^{-1})	Ring crush index (N m g^{-1})	Burst index (kPa m^2 g^{-1})	Cobb value (g m^{-2})
1		34.8	3.5	1	127
2	6.3	53.9	5.6	1.7	92
3	6.2	48.5	5.5	1.5	35.2
4	6.4	46.2	5.9	1.5	39.8

1 Original paper, *2* 61 % Starch +19 % Hemicellulose +20 % $Al_2(SO_4)_3$, *3* 72 % Starch +8 % Lignin +20 % $Al_2(SO_4)_3$, *4* 78.8 % Starch +1.2 % Extractives +20 % $Al_2(SO_4)_3$

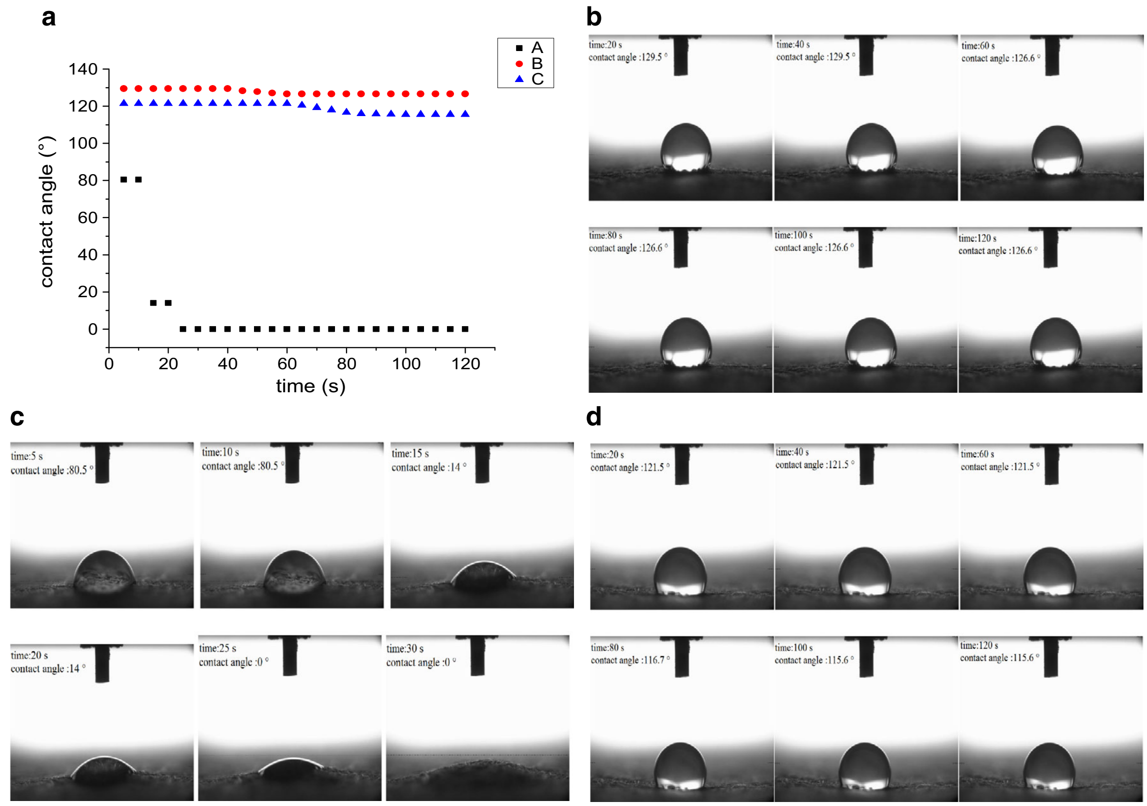

Fig. 4 Dynamic contact angles of microscopic droplets of water on corrugated paper vs time. **a** *A* Original paper, *B* 72 % Starch +8 % Lignin +20 % $Al_2(SO_4)_3$, *C* 78.8 % Starch +1.2 % Extractives +20 % $Al_2(SO_4)_3$. **b** Dynamic contact angles of the corrugated paper sized by sizing agent containing lignin. **c** Dynamic contact angles of original corrugated paper. **d** Dynamic contact angles of the corrugated paper sized by sizing agent containing extractives

Figure 4 shows the observed variation of contact angle of the corrugated paper sized by different components of APMP effluent at different contact time. The water contact angles of original corrugated paper show a definite reduction as the time is increased. Water droplet spreads excessively on the paper in 30 s. However, the water contact angles of corrugated paper, sized by the sizing agent contained lignin and extractives, are substantially unchanged with increasing time. The water contact angles were 126.6° and 115.6° at 120 s, respectively, the nature of which reflect is consistent with the results in Table 5.

The above experiments demonstrate that the sizing agents [APMP effluent combined with starch and $Al_2(SO_4)_3$] could improve the water resistance of corrugated paper. Lignin and extractives combined with Al^{3+} play a major role in hydrophobic properties of corrugated paper. The hydrophilic groups (–OH) (–COO–) are combined with the paper fibers by Al^{3+} in the sizing process. At the same time, the hydrophobic groups (phenylpropane and C14–C20) can stretch orient to face outwards and form a continuous film layer, which tends to be water-repellent.

Conclusions

Corrugated paper sized by sizing agent (Starch:APMP effluent:$Al_2(SO_4)_3$ = 2:2:1) had low Cobb value 22 g/m^2, which showed good water resistance. The water resistance mechanism indicated that lignin and extractives combining with Al^{3+} played a major role in hydrophobic properties of corrugated paper. The sizing agents containing lignin and extractives could reduce the Cobb value to 35.2 and 39.8 g/m^2, respectively. The water contact angles were 126.6° and 115.6° at 120 s, respectively. The result of GC/MS analysis of APMP effluent showed fatty acids (C14–C28) and their esters, sterols were the main components of APMP effluent extractives.

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