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Coagulation and Adsorption Treatment of Printing Ink Wastewater

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Abstract:

The intention of the study was to improve the efficiency of total organic carbon (TOC) and colour removal from the wastewater samples polluted with flexographic printing ink following coagulation treatments with further adsorption onto activated carbons and ground orange peel. The treatment efficiencies were compared to those of further flocculation treatments and of coagulation and adsorption processes individually. Coagulation was a relatively effective single-treatment method, removing 99.7% of the colour and 86.9% of the organic substances (TOC) from the printing ink wastewater samples. Further flocculation did not further eliminate organic pollutants, whereas subsequent adsorption with 7 g/l of granular activated carbon further reduced organic substances by 35.1%, and adsorption with 7 g/l of powdered activated carbon further reduced organic substances by 59.3%. Orange peel was an inappropriate adsorbent for wastewater samples with low amounts of pollution, such as water that had been treated by coagulation. However, in highly polluted printing ink wastewater samples, the adsorption treatment with ground orange peel achieved efficiencies comparable to those of the granular activated carbon treatments.

Keywords:

Activated Carbon, Adsorption, Coagulation, Orange Peel, Printing Ink, Wastewater

1. Introduction

Approximately 60–70% of typical flexographic printing ink consists of solvents, such as alcohols, acetates and glycol ethers. Organic solvents, nearly all of which are flammable volatile organic compounds (VOCs), are irritants and health risks. Furthermore, they contribute to the formation of tropospheric ozone and smog, both of which cause respiratory diseases. In water-based inks, the majority of the organic solvents are replaced with water. Water-based inks are suitable for flexographic printing on paper and paperboard and cause only low VOC emissions to the surrounding air during print drying. Therefore, they are more environmentally favourable than inks that are based solely on organic solvents.

Nevertheless, water-based inks can also become an ecological problem when they remain in wastewater after printing plates and printing press cylinders are cleaned with tap water. Such wastewater is overloaded with organic substances that are generally not biodegradable and are dangerous to aquatic life. Therefore, the flexographic printing industry must treat this type of wastewater prior to discharge into the aquatic environment with methods that are, if possible, efficient and inexpensive.

Some information can be found in the literature on the treatment of printing ink wastewater, including coagulation with aluminium and ferric salts (Meteš, et al., 2000), resulting in 66–85% removal of total organic carbon (TOC) and 80–92% removal of chemical oxygen demand (COD); coagulation with polyaluminium chloride (Nandy, et al., 2002), resulting in 58% removal of COD; treatment with aluminium sulphate and polyacrylamide (Ma, et al., 2010), resulting in a maximum COD reduction of 90%; coagulation and adsorption using synthetic zeolite (ZSM-5) (Meteš, et al., 2004), resulting in 95% removal of TOC; coagulation and flocculation with chitosan and tannin (Roussy, et al., 2005), resulting in 84% removal of COD; chemical oxidation using the Fenton process and coagulation (Ma, et al., 2009), resulting in 93.4% COD removal; biological treatment with Bacillus sp. Bacteria (Zhang, et al., 2003), resulting in approximately 85% COD removal and coagulation and electrochemical oxidation (Diamadopoulos, et al., 2009), resulting in 60% COD removal.

The objective of this study was to improve the efficiency of the treatment of wastewater that was polluted with flexographic printing ink using a combination of coagulation and adsorption onto commercially available activated carbons in both powdered and granular forms and onto ground orange peel obtained from domestic waste. The prevailing methods for the treatment of coloured effluents, coagulation and flocculation, were performed, and the effects of the combination of the two methods were compared to coagulation in combination with adsorption and to adsorption alone.

A preliminary study on the effectiveness of different inorganic and organic coagulants in similar printing ink wastewater samples showed that the best treatment results were obtained using inorganic trivalent iron and aluminium salts (Klančnik and Židanik, 2007). Due to the possible impact of aluminium on the development of Alzheimer's disease, ferric salts are preferred. In this investigation, the different ionic natures of the commercial flocculants also revealed that only anionic polymers increased the formation of flocs in water that had been treated with coagulation. Therefore, in the present work, iron (III) chloride was chosen as the coagulant, and commercial anionic polyacrylamide was chosen as the flocculant to destabilise the ink suspensions and encourage the formation of flocs.

Due to its extremely high specific surface area, activated carbon is the most widely used adsorbent for the removal of organic contaminants from air or water through physical adsorption. Numerous studies have been conducted on the adsorption of different classes of soluble textile dyes in wastewater effluents onto activated carbon (Reife and Freeman, 1996; Papić, et al., 2004; Suteu and Bilba, 2005; Lee and Choi, 2006). Orange peel is an example of a natural, biodegradable and inexpensive adsorbent. It has already proven efficient for the elimination of different classes of textile dyes from aqueous solutions in which the concentrations of the dyes were monitored using absorbance spectrophotometry (Arami, et al., 2005; Ardejani, et al., 2007; Kumar and Porkodi, 2007). In this investigation, however, the removal of commercial printing ink, which is a complex suspension of insoluble pigment particles, solvents, soluble resins and other additives, was investigated. The treatment efficiency in wastewater was assessed using TOC analyses to measure the removal of dissolved and suspended organic matter and spectrophotometric analyses to measure colour removal.

2. Materials and Methods

2.1 PRINTING INK WASTEWATER

Two wastewater samples were prepared – one with Aquanol Red, a commercially available red water-based flexographic printing ink, and the other with Aquanol Special Blue, a commercially available blue water-based flexographic printing ink. Both inks were produced by Cinkarna Celje (Slovenia). Each wastewater sample contained 2 g of printing ink in 1 l of tap water. The tap water had hardness of 20°n and pH value of 7.40.

2.2 COAGULANT

For the coagulation process, a 10% solution of ferric (III) chloride ($FeCl_3 \cdot 6H_2O$), produced by Riedel-de Haën, was prepared in double-distilled water.

2.3 FLOCCULANT

The commercial product CHT-Flocculant ACL (CHT), based on anionic modified polyacrylamide, was used as a 0.05% solution and was prepared with double-distilled water.

2.4 ADSORBENTS

Powdered activated carbon (PAC) and granular activated carbon (GAC) were both produced by Riedel-de Haën. The PAC particle sizes ranged from 1.25-158 µm, and the GAC particle sizes ranged from 0.516-2.78 mm.

Orange peel was obtained from domestic waste. It was first air-dried at room temperature for several days and was then ground with a domestic coffee grinder into powdered form with particle sizes ranging from 0.034-1.61 mm.

The sizes of the adsorbent particles were determined using a JEOL 6060 LV scanning electron microscope (SEM) (JEOL, Japan).

2.5 TREATMENT METHODS

Coagulation, flocculation and adsorption were conducted as jar (batch) tests in a laboratory rotational mixer/flocculator (Fisher Bioblock Scientific) that was equipped with six flat paddles. The initial pH values of the wastewater samples were not adjusted prior to the treatments. For each treatment, at least two replicate experiments were performed.

Prepared wastewater samples (200 ml) in six beakers were first mixed with different amounts of ferric solution to determine the optimal addition of coagulant. The coagulation process was performed with 2 min of rapid stirring (150 rpm), followed by 5 min of slow stirring (30 rpm).

Afterwards, different amounts of flocculant were added to the 200 ml samples of optimally coagulated water samples, and the flocculation process proceeded with 5 min of slow stirring (30 rpm).

Different amounts of activated carbon (1, 2, 3, 4, 5, 6, and 7 g/l) and orange peel (1, 3, 5, and 7 g/l) were assessed for optimal adsorption using 200 ml of optimally coagulated wastewater with flocs still present. The adsorption treatment process was performed at 23°C with 60 min of constant stirring (80 rpm) and one day of adsorbent settling. The adsorption experiments were also performed with 7 g/l of adsorbent and 200 ml of polluted wastewater as a single-treatment process.

Before and after each experiment, the pH was measured with a pH meter (315i, WTW). After one day of sedimentation, supernatant water samples were acquired and spectrophotometrically and TOC analysed without filtering. One day of sedimentation was practically chosen for withdrawing the samples for measurements, because the flocs after flocculation and as well as the adsorbents after adsorption were well separated from the rest of the wastewater. Untreated wastewater was also for comparison spectrophotometrically and TOC analysed.

Different concentrations of orange peel (1, 3, 5, and 7 g/l) were added to tap water to determine the colour and TOC contributions of the orange peel. These samples were filtered through filter paper one day after preparation and prior to analysis.

The TOC was determined using a Shimadzu 5000A TOC analyser (Shimadzu Co). The colour of the wastewater was determined as a spectral absorption coefficient (SAC) at the wavelength of maximum absorption, which is absorption per unit optical length, using a Cary 1E UV-VIS transmittance spectrophotometer (Varian).

3. Results and Discussion

Both wastewater samples were slightly alkaline (pH 7.9), therefore, neutralisation was not required. The characteristics of the prepared flexographic printing ink wastewater samples are shown in Tables 1 and 2. The wastewater samples were strongly coloured and were heavily polluted with organic substances, which is reflective of industrial printing wastewater. These wastewater samples cannot legally be discharged directly into surface water or into public wastewater systems (cf., Official Gazette RS, 64/2012). Both water-based printing inks contained the same concentrations of the same types of organic solvents, i.e., less than 20% of ethanol and isopropanol (Material safety data sheet, 2000). The suspensions of the same concentrations of red and blue printing inks initially had different SAC and TOC values, as they contained different chemical structures, slightly different amounts of pigments (approximately 10-15%) and different acrylic or melamine resins as binders (15-20%). The other additives in the printing inks were defoaming agent and waxes, which were present in very small concentrations (1-5%).

The efficiency of the treatment processes was evaluated as removal of organic substances (TOC value) from printing ink wastewaters as well as removal of colour, i.e., the SAC value at the wavelength of maximal absorption. However, conventional printing inks are suspensions of insoluble pigment particles. Therefore, absorption spectrophotometry is not an objective method for the evaluation of actual treatment efficiencies. The SAC measurements were necessary only to evaluate the decolourisation effects of the treatment. The TOC analysis is a much more appropriate method, as it provides the concentrations of total organic carbon that are bound to the dissolved and suspended organic matter in the wastewater. The efficiencies of TOC removal after different treatment methods are shown in Figure 1 for the red printing ink wastewater and in Figure 2 for the blue printing ink wastewater.

The coagulation process was successful, achieving high colour removal (approximately 99.7%) and organic substance removal (89% and 84.9% in the red and blue printing ink wastewater samples, respectively). It was found out, that the optimal coagulant doses were 3.25 ml/l for the red ink and 2.75 ml/l for the blue ink wastewater. During the coagulation process, a significant amount of positively charged ferric hydrolysis products and relatively low concentration of free trivalent ferric ions (Fe^{3+}) from the coagulant (Black, 1960; Stumm and Morgan, 1962) neutralised the negatively charged colloidal printing ink particles (Lee, 2000), causing the formation of numerous small agglomerates in the wastewater. The agglomerates completely settled out within 15 minutes after stirring was ended. Following coagulation, the solution of the initially red printing ink wastewater was clear and only slightly coloured, while the initially blue printing ink wastewater was colourless. However, the TOC wastewater values after coagulation still exceeded the maximum discharge allowance (30 mg C/l, cf., Official Gazette RS, 64/2012). To achieve an additional reduction of TOC, further flocculation and adsorption processes were investigated.

During the flocculation process, anionic polyacrylamide attracted the cationic agglomerate particles from the coagulated wastewater and bound them into larger flocs. These were quickly separated from the water after only 5 minutes, which then became clear and colourless. The optimal doses of the 0.05% solution of commercial flocculant were 1.1 g/l in the red printing ink wastewater and 0.75 g/l in the blue printing ink wastewater. The wastewater that was treated with the combination of ferric (III) coagulant and anionic flocculant (see Tables 1 and 2) had slightly lower SAC value and essentially no additional reduction in TOC compared to the wastewater that was treated with coagulation only.

When adsorption with activated carbon was performed after the optimised coagulation process, the treated wastewater samples were colourless and had sufficiently low SAC values. Adsorption is a very complex process in which various printing ink ingredients are adsorbed onto the surface of the activated carbon. The TOC value in the red printing ink wastewater was low enough for discharge into surface water after coagulation and further adsorption with 2 g/l of PAC, with 94.9% TOC elimination (see Table 3). After adsorption with the highest concentration of PAC (7 g/l), the organic compound removal efficiency additionally increased by 71.8% (the total efficiency of coagulation and PAC adsorption was 96.9%) in the red ink wastewater and by 46.8% (the total efficiency was 91.9%) in the blue ink wastewater. Nevertheless, the blue printing ink wastewater still did not have sufficiently low TOC value (cf. Table 4). The blue printing ink wastewater required further treatment, for example, with chemical oxidation or biological degradation.

During adsorption with 7 g/l of GAC, an additional 37.7% and 32.5% of the organic compounds were removed from the coagulated red and blue printing ink wastewater, respectively (see Tables 5 and 6). Treatment with GAC resulted in approximately the same additional reduction in organic substances in both polluted wastewater samples. The total efficiency of TOC removal with combination of coagulation and GAC adsorption was 93.5% and 89.8% for the red and for the blue printing ink wastewater, respectively. GAC exhibited an adsorption capacity that was approximately 2.7 times lower than the PAC. The activated carbon in the granular form has much larger particle sizes than PAC and presents a smaller external surface area, consequently adsorbing smaller amounts of organic contaminants than PAC. Nevertheless, GAC was still more efficient at organic compound removal than the polymer flocculant. While GAC is more easily separated from treated water, PAC settled very well after one day of sedimentation, and the water samples required for the analysis were easily collected above the sediment layer.

Table 1. - TOC and SAC results of wastewater contaminated with Aquanol Red printing ink before and after coagulation and flocculation treatment

Parameters	Wastewater before treatment	Wastewater after coagulation	Wastewater after coagulation and flocculation
pH at 23°C	7.90	7.37	7.20
SAC at 569 nm (m–1)	3625.70	5.39	3.42
TOC (mg C/l)	547.15	60.23	59.27

Table 2. - TOC and SAC results of wastewater contaminated with Aquanol Special Blue printing ink before and after coagulation and flocculation treatment

Parameters	Wastewater before treatment	Wastewater after coagulation	Wastewater after coagulation and flocculation
pH at 23°C	7.89	7.40	7.20
SAC at 629 nm (m–1)	526.75	1.95	1.47
TOC (mg C/l)	498.96	75.55	75.53

Table 3. - TOC and SAC results of wastewater contaminated with Aquanol Red printing ink after coagulation and adsorption with different concentrations of PAC and after adsorption alone

Concetration of orange peel								
Parameters	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l	6 g/l	7 g/l	7 g/l without coagulation
pH at 23°C	7.01	7.01	7.01	7.05	7.05	7.05	7.05	8.3
SAC at 569 nm (m–1)	2.41	2.38	2.05	1.71	1.45	1.04	1.01	2602.32
TOC (mg C/l)	30.39	27.72	27.68	26.47	25.19	18.51	16.96	380.80

Table 4. - TOC and SAC results of wastewater contaminated with Aquanol Special Blue printing ink after coagulation and adsorption with different concentrations of PAC and after adsorption alone

Concetration of orange peel								
Parameters	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l	6 g/l	7 g/l	7 g/l without coagulation
pH at 23°C	7.7	7.7	7.7	8.0	8.0	8.1	8.1	8.6
SAC at 629 nm (m–1)	2.89	1.15	0.87	0.74	0.68	0.64	0.57	527.05
TOC (mg C/l)	59.37	56.09	50.88	48.77	45.76	45.02	40.22	330.15

Table 5. - TOC and SAC results of wastewater contaminated with Aquanol Red printing ink after coagulation and adsorption with different concentrations of GAC and after adsorption alone

Concetration of orange peel								
Parameters	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l	6 g/l	7 g/l	7 g/l without coagulation
pH at 23°C	7.34	7.44	7.45	7.45	7.46	7.49	7.49	8.20
SAC at 569 nm (m–1)	1.73	3.16	3.57	3.82	3.91	4.88	4.96	3625.94
TOC (mg C/l)	57.11	52.55	48.35	44.82	41.97	39.47	37.50	528.50

Table 6. - TOC and SAC results of wastewater contaminated with Aquanol Special Blue printing ink after coagulation and adsorption with different concentrations of GAC and after adsorption alone

Concetration of orange peel								
Parameters	1 g/l	2 g/l	3 g/l	4 g/l	5 g/l	6 g/l	7 g/l	7 g/l without coagulation
pH at 23°C	7.3	7.1	7.7	7.6	7.65	7.7	7.7	8.3
SAC at 629 nm (m–1)	1.32	0.94	0.92	1.11	1.69	1.44	1.82	527.03
TOC (mg C/l)	72.79	68.25	61.84	58.74	54.45	52.27	50.99	418.90

Although ground orange peel has proven to be efficient in removing dyes from water (Arami, et al., 2005; Ardejani, et al., 2007; Kumar and Porkodi, 2007), this was not observed in the coagulated printing ink wastewater samples. The colour intensities increased slightly with increasing orange peel concentrations, and the pH values decreased to below the acceptable pH range (6.5-9, cf., Official Gazette RS, 64/2012), as shown in Tables 7 and 8. The biggest problem with the use of orange peel was its contribution to organic pollution, which was revealed by the TOC analyses. The TOC values increased considerably with increasing orange peel doses (see Table 9). As a result, the TOC values of the wastewater samples after treatment with orange peel (see Tables 7 and 8) were much higher than those of the initial printing ink wastewater (Tables 1 and 2). Therefore, during the calculation of the TOC removal efficiency from adsorption using ground orange peel, the initial TOC values were modified by adding the TOC value of orange peel itself (Table 9) to the initial TOC values of the wastewater samples before treatment (Tables 1 and 2). Therefore, the efficiency of organic matter removal from the coagulated wastewater samples decreased with increasing orange peel concentrations (see Tables 7 and 8).

The adsorption process with 7 g/l of PAC without prior coagulation removed only 30.4% and 33.8% of the organic impurities from the red and blue printing ink wastewater, respectively (Tables 3 and 4, Figures 1 and 2). Using 7 g/l of GAC, only 3.4% and 16.1% of the organic impurities were removed from the red and blue printing ink wastewater, respectively (Tables 5 and 6, Figures 1 and 2). Adsorption with activated carbon as a single-treatment process did not eliminate any colour; therefore, the wastewater samples remained turbid and highly coloured after the adsorption treatment, visually appearing the same as before treatment.

Table 7. - TOC and SAC results of wastewater contaminated with Aquanol Red printing ink after coagulation and adsorption with different concentrations of orange peel and after adsorption alone

Concetration of orange peel					
Parameters	1 g/l	3 g/l	5 g/l	7 g/l	7 g/l (w/o coagulation)
pH at 23°C	6.45	5.15	4.70	4.32	4.45
SAC at 569 nm (m–1)	3.48	3.21	5.72	6.51	3623.03
TOC (mg C/l)	259.75	656.35	1014.9	1522.5	2066.4

Table 8. - TOC and SAC results of wastewater contaminated with Aquanol Special Blue printing ink after coagulation and adsorption with different concentrations of orange peel and after adsorption alone

Concetration of orange peel					
Parameters	1 g/l	3 g/l	5 g/l	7 g/l	7 g/l (w/o coagulation)
pH at 23°C	6.62	5.40	4.80	4.37	4.40
SAC at 629nm (m–1)	1.78	3.43	3.48	2.07	431.89
TOC (mg C/l)	280.2	660.7	991.58	1523.5	1869.0

Table 9. - TOC and SAC results of tap water contaminated with different concentrations of orange peel

Concetration of orange peel				
Parameters	1 g/l	3 g/l	5 g/l	7 g/l
SAC at 569 nm (m–1)	0.44	2.28	6.62	8.89
SAC at 629 nm (m–1)	0.22	1.73	5.30	7.01
TOC (mg C/l)	231.60	647.38	989.75	1521.8

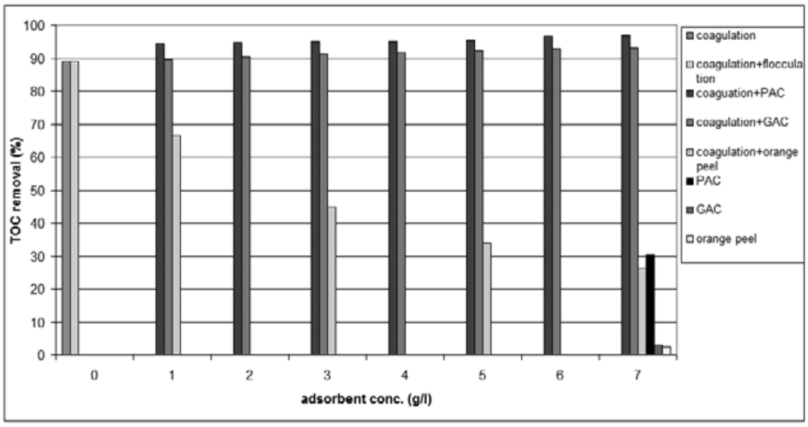

Figure 1. Efficiency of TOC removal from Aquanol Red printing ink wastewater with coagulation, flocculation and adsorption with different concentrations of powdered activated carbon (PAC), granular activated carbon (GAC) and orange peel

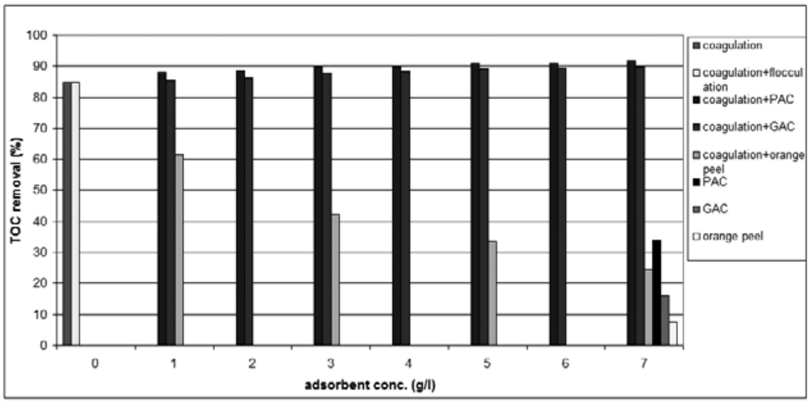

Figure 2. Efficiency of TOC removal from Aquanol Special Blue printing ink wastewater with coagulation, flocculation and adsorption with different concentrations of powdered activated carbon (PAC), granular activated carbon (GAC) and orange peel

Orange peel, surprisingly, exhibited slight adsorption in non-coagulated, highly ink-polluted wastewater samples, where a concentration of 7 g/l removed 2.5% and 7.5% of the organic matter from the red and blue printing ink wastewater, respectively (Figures 1 and 2), and it also removed some of the colour (18%, Table 8) from blue printing ink wastewater. Because of the low pH values and the very high TOC values of the wastewater samples that contained the orange peel, these wastewater samples required neutralisation and further biological treatment. Orange peel was not an appropriate adsorbent for the lightly polluted wastewater, such as the water that had been pretreated with coagulation, with TOC values at approximately 60-76 mg C/l. However, in highly polluted printing ink wastewater, with TOC values of approximately 547-499 mg C/l, the adsorption treatment with ground orange peel achieved only a little lower TOC removal efficiencies than treatment with GAC.

4. Conclusion

The coagulation of simulated flexographic printing ink wastewater was a very effective method for the elimination of colour (99.7%) and organic substances (86.9%). An additional reduction of organic substances was not achieved with subsequent polymer flocculation but was achieved with subsequent adsorption treatment using activated carbon. As it was expected, PAC was much more effective at removing TOC than GAC, as it eliminated on average 46.8% of the organic contaminants from the previously coagulated wastewater, compared to 22.2% for GAC. In the heavily polluted printing ink wastewater, PAC only removed approximately 32.1% of the organic substances and almost no colour.

Orange peel, which was not an appropriate adsorbent in previously coagulated wastewater, showed little adsorption efficiency in highly polluted printing ink wastewater, removing on average 5% of organic matter with efficiencies comparable to those of GAC (9.7%).

The combination of coagulation with ferric (III) chloride and adsorption with PAC was the most efficient treatment method for the printing ink wastewater. The process successfully decolourised and reduced organic impurities (TOC) in red printing ink wastewater (by 94.5–96.9%) and blue printing ink wastewater (by 88.1–91.9%). Despite the failure of the combined coagulation and adsorption to eliminate sufficient levels of organic contaminants from the blue ink wastewater to allow for direct discharge into surface water, the treated wastewater could be released into the public sewage system, where aerobic biological treatment is conducted. Treated wastewater can also be reused for the cleaning of printing equipment, which would reduce not only the amount of discharged industrial wastewater but also the consumption of fresh water.

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