Materials Transactions, Vol. 44, No. 2 (2003) pp. 313 to 319

Use of Kerosene to Improve Toner-Ink Liberation for Office Paper Recycling*1

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A method to disintegrate photocopied or laser printed waste office paper by weak agitation and using kerosene as a toner softening agent was investigated. Under the conventional method using only sodium hydroxide, 3.8% of toner was liberated from paper. This figure increased to 98.1% by using undiluted kerosene, and to 80.1% by using 1% kerosene emulsion. Residual toner particles adhered to only one or a few paper fibers after these kerosene treatments. If residual toner was also included in this figure, toner liberation would reach 100%. The use of surfactants as dispersion agents for kerosene decreased the degree of liberation because it interferes with the softening of toner. It was also confirmed that sodium hydroxide treatment in advance of the kerosene treatment is important to increase toner liberation.

(Received June 21, 2002; Accepted November 8, 2002)

Keywords: *paper recycling, office paper, toner, kerosene, liberation, deinking*

1. Introduction

The production of office paper in Japan has increased about 300% during the past decade. Most of this paper is of a high grade and very bright, and can be recycled into other high-grade paper. However, the recycling percentage of office paper has not increased with production because the deinking process involved is more difficult than that for conventional waste paper such as newsprint and magazines.[1)] Oil-based ink can be liberated easily from the fiber in conventional waste paper by saponification in alkali solution. However, toner ink is used instead of oil-based ink in almost all office waste paper generated by photocopying machines and laser printers. Toner is a solid particle that consists of many sub-micron pigments covered with resin.[2)] It is melted into fibers like an anchor when being printed, but resin cannot be saponificated in the deinking process. Therefore, it is difficult to liberate toner particles from paper fibers by the conventional deinking process, and they remain locked in the fibers.[3,4)] In the recycling of office paper, these remaining toner particles, called "toner specks," affect the value of regenerated paper as well as paper whiteness.[5,6)] Toner specks are visible black particles over 30 μm in size that remain on regenerated paper. Their existence makes the distribution of regenerated paper difficult, even if it shows high whiteness.

Under such circumstances, many studies on the disintegration of waste office paper have been reported. Most studies have investigated methods such as strong agitation,[3,7,8)] ultrasonic wave irradiation,[9,10)] and enzymatic treatment[11)] to reduce the size of the particles in order to achieve greater toner liberation. However, it would be difficult to use the fibers that result from these methods to make high grade regenerated paper because they would be shredded if the waste paper comminuted until the toner particles can be liberated, which occurs at a size of approximately 10 μm. Therefore, incomplete liberation is inevitable at present. To liberate toner without cutting the fibers, it is necessary to separate the toner and fiber at their interfaces by weak agitation. As the main composite of toner is thermoplastic resin, one might consider that liberation would be made easier by heating it, thereby softening the toner. But the fact that the maximum softening temperature of toner is around 120°C makes heating it difficult because the normal deinking process is done in water; that is, it is impossible to raise the temperature of water to this point. Although a heating method in the dry atmosphere has been developed to reuse office paper, this has not been extended to general use due to paper deterioration and because the softening temperatures change with particular types of toner.[12,13)]

On the other hand, the resin in toner is known to soften and dissolve in various kinds of organic solvents.[3)] Therefore, it may be possible to liberate toner by weak agitation at ambient temperature if it can be softened by those solvents. While the use of solvents in waste paper regeneration studies for the purpose of dissolving stickies (*e.g.*, 3M Post-It® notes) and alkali insoluble components of conventional waste paper has been reported,[3,6)] there are very few examples using solvents to soften toner in order to increase toner liberation. In this paper, a new disintegration method was investigated to achieve high toner liberation for waste office paper by weak agitation at ambient temperature using kerosene, which is low in price and widely used as a solvent.

2. Experimental

2.1 Sample

Toner is classified into two types: magnetic and non-magnetic, based on whether or not it contains magnetic particles. Styrene-acryl resin is widely used for toner resins.

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*1This Paper was Published in Japanese in Journal of the Mining and Materials Processing Institute of Japan **118**, No. 3, 4 (2002).
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The use of high transparency polyester resin has increased recently with the spread of colored toner. Taking into account these toner components, magnetic toner that uses styrene-acryl resin (Canon: CRG-EPKS II, resin content: about 40%) and non-magnetic toner that uses multiple resin containing polyester resin (Fuji-Xerox: D282, resin content: about 90%) were chosen as toner samples in this study. A laser printer that uses magnetic toner (Canon: Laser Shot B406E) and a photocopying machine that uses non-magnetic toner (Fuji-Xerox: Vivace 720) were utilized. The printed samples were prepared by printing 100 black circles (11 point font) on one side of a piece of photocopy paper (NBC-Ricoh: 90-2218, 65 g/m^2) with the laser printer using magnetic toner. Next, a copy of this printed paper was made with the photocopying machine using non-magnetic toner. Then, these two pieces of printed paper were cut into 50 mm × 50 mm pieces (Fig. 1) and used as printed samples for experimentation. The amount of the area printed was 33.6% for the magnetic and 30.9% for the non-magnetic samples. The reason black circles were printed on the samples instead of characters, which are typically printed on waste office paper, is because they allow for easier measurement of toner liberation. Moreover, deinking one particular area is more difficult than one character.[14] Figure 2 shows the size distribution in cumulative volume of each toner sample before being printed, which was calculated from the Heywood diameter measured by a continuous flow image analyzer (Nireco: Luzex SE-SPL). Measurements were carried out for each toner suspension, which was prepared by placing 0.5 g of toner sample into 2000 mL of de-ionized water, adding 4 g of sodium hexametaphosphate, and irradiating it with an ultrasonic wave for 5 min. Ten thousand particles were measured in each suspension. The 50% diameters by volume of both samples were 8.7 µm for the magnetic and 15.3 µm for the non-magnetic sample.

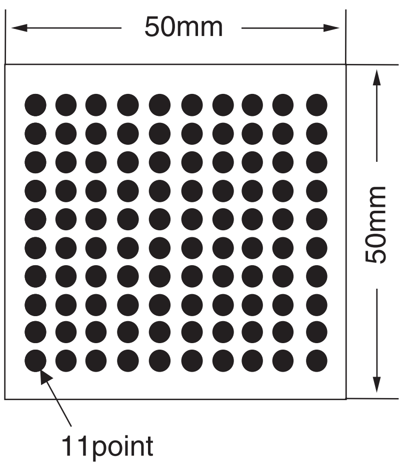

Fig. 1 Illustration of model waste paper sample.

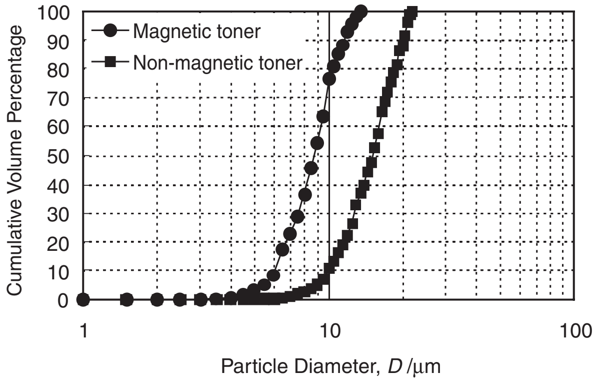

Fig. 2 Size distribution of toner samples.

2.2 Experimental methods

2.2.1 Observation of toner softening

After adding 1 mL of kerosene or other solvents with 0.1 mL of surfactant (22% Sodium alkyl ether sulfate) to de-ionized water, the total volume was adjusted to 100 mL with de-ionized water. This was emulsified by stirring with a disperser (Yamato: LK-21) at 333.3 s^{-1} for 1 min. A piece of printed sample paper (0.15 g) was soaked in 100 mL of 8% sodium hydroxide solution for 12 h and then washed with de-ionized water. This sample was put into the emulsion and stirred by a stainless impeller (rotation diameter: 50 mm) at 2 s^{-1} for 30 min, then washed again. The paper did not disintegrate under this stirring condition. The softening state of the printed sample was observed with a microscope (Nikon: SMZ-1) without drying it. When experiments on changing the order of sodium hydroxide and kerosene soaking were conducted, the sample was soaked with sodium hydroxide solution after being soaked with kerosene.

2.2.2 Softening degree of toner

The degree of toner softening was measured by a tackiness tester (Malcom: TK-1) to investigate the softening efficiency of kerosene. Sample tablets 10 mm in diameter and 3 mm in thickness were made by pressing 0.3 g of each magnetic and non-magnetic toner sample with a hydraulic press at 20 MPa for 5 min. Each sample tablet was glued onto the bottom of a 25 mL weighing bottle, which was set to the holder of the tackiness tester. Then, 10 mL of kerosene emulsion at several different concentrations was added to the weighing bottle. The tablet sample was pressed by a column-shaped stainless probe (ϕ 5.1 mm) at 3.92 N for 0.2 s. The toner's degree of softening was measured by the pulling stress at a pull speed of 2.0 mm/s. The measurements were carried out at ambient temperature (20°C), and were basically conducted following JIS standard Z3284, which provides an adhesion measurement for solder paste. However, a higher press load and lower pulling speed than what is considered to be standard was set because the pulling stress of toner is very weak.

2.2.3 Degree of toner liberation

After disintegrating the paper sample printed with magnetic toner, it was possible to recover all toner by magnetic separation. The degree of liberation was then measured using a microscope by observing whether or not each toner particle had bonded to the paper fibers. A piece of sample paper printed with magnetic toner was soaked in 100 mL of 8% sodium hydroxide (pulp concentration 0.15%) for 12 h in order for it to swell. Then, the sample was stirred by an impeller at 2 s^{-1} for 30 to 180 min in several different concentrations of a 100 mL kerosene emulsion. When surfactants (shown in 2.2.1) were added to the emulsions, the same kind of stirring was also carried out. After soaking the sample in kerosene it was recovered by filtration with a No. 5A filter. The recovered sample was placed in 100 mL of 8% sodium hydroxide solution and stirred with an impeller at $8.3\,s^{-1}$ for 5 min for disintegration. This stirring condition was decided upon from preliminary tests as the minimum stirring power required for paper disintegration. Next, this disintegrated sample was poured into a 300 mL beaker where all the toner particles were recovered with a neodymium-boron magnet. The recovered particles were placed on a microscope, the images from which were fed into a personal computer through a video camera (Victor: KY-F57). From these images, the total area of both the liberated toner and locked toner (toner particles bonded to the paper fibers) were measured using image analysis software (Mitani: Winroof), and the ratio of the area of liberated toner to the entire toner area (liberated + locked) was calculated as the degree of liberation. The size of the area was adopted here because the shape of almost every speck of separated toner was flat.[15,16]

3. Results and Discussion

3.1 Observation of toner softening

Toner softening states in kerosene emulsions were observed by the method described in 2.2.1. The microscopic observation results are shown in Table 1 and Figs. 3(a) and (b). Results of the toner softening by the use of other representative solvents for resin (1% emulsion in each) are also described in Table 1. All solvents, including kerosene, softened the toner on the printed samples well. But when observed in detail, the softening states varied among the combinations of solvents and type of toner. When kerosene was used, magnetic toner was softened and could be peeled off easily from the paper's surface with tweezers (Fig. 3(a)). In non-magnetic toner samples, the stickiness of the toner increased along with the softening process, and roped before being peeled off with tweezers (Fig. 3(b)). Toner printed using electrophotographic systems infiltrated the paper fibers and solidified such as in Fig. 4(a). The infiltrated part wedged into the paper fiber is referred to as the ''anchor effect'' and is one of the reasons behind the difficulty of toner deinking.[3,12] Since no wedged parts remained on the paper fibers after picking at the toner with tweezers, it can be said that the wedged parts were peeled off altogether as shown in Fig. 4(b). When n-heptane was used the softened state of toner was the same as that with kerosene. Although toner was also softened well with toluene or xylene, some resin dissolved and the emulsion turned muddy gray in color. This phenomenon suggests that fine pigment particles (diameter of about 100 nm) disperse into the emulsion by resin dissolution, which may decrease paper whiteness. Generally, aromatic solvents such as toluene and xylene have a higher dissolving ability for toner resin than aliphatic solvents such

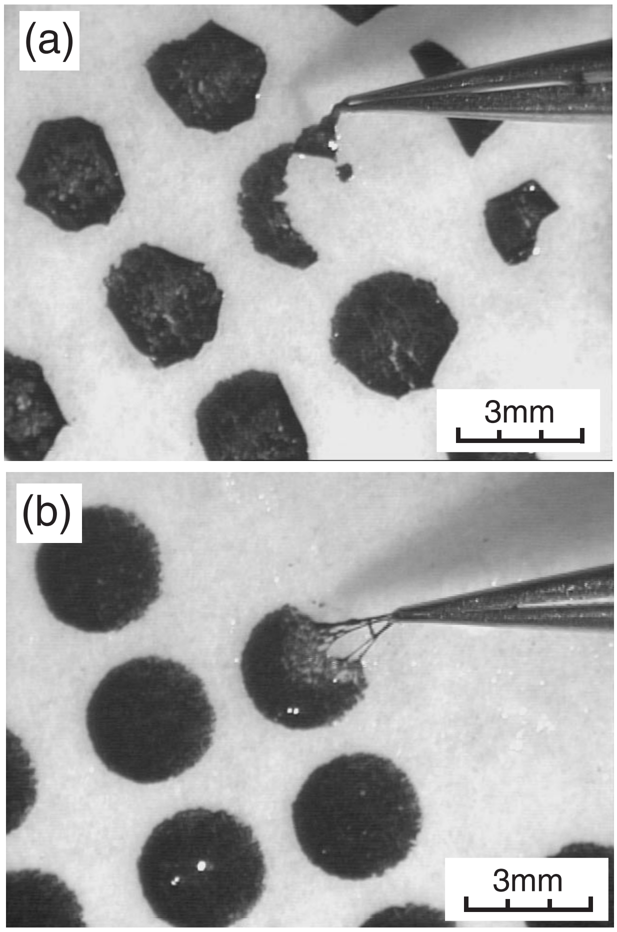

Fig. 3 Toner picking test after softening by kerosene. (a) Magnetic toner sample (b) Non-magnetic toner sample.

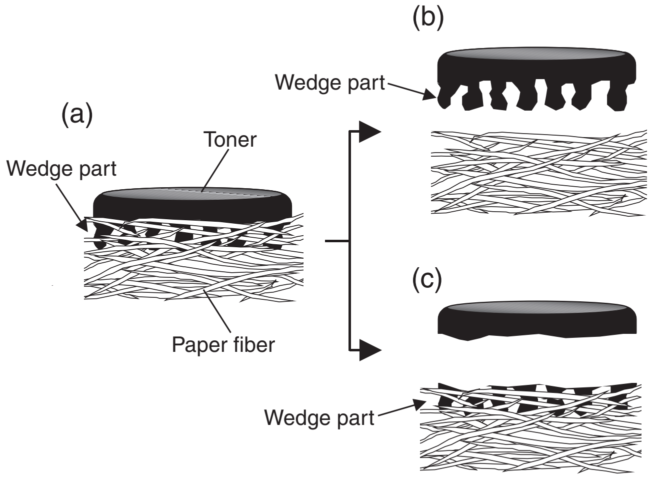

Fig. 4 Illustration of toner treated with kerosene. (a) Anchor effect of wedge part in printed state (b) Wedge part peeled off by picking (c) Remained wedge part.

Table 1 Softening of toner by kerosene and other solvent emulsions.

Regents	Formula	Magnetic toner sample	Non-magnetic toner sample
Kerosene	C_nH_{2n+2}	Softens well	Softens well and shows some adhesion
n-Heptane	$CH_3(CH_2)_5CH_3$	Softens well	Softens well and shows some adhesion
Toluene	$C_6H_5CH_3$	Softens well and dissolves partially	Softens well and dissolves partially
Xylene	$C_6H_4(CH_3)_2$	Softens well and dissolves partially	Softens well and dissolves partially

as kerosene and n-heptane,[3,17,18] but the power of these aromatic solvents seems to be too strong for the softening of toner when the purpose is to increase the degree of liberation. From the results of these experiments, there is nothing that distinguishes kerosene and n-heptane. However, it can be said that kerosene is an effective and reasonable solvent as a softening reagent for toner because the price of kerosene is lower than that of n-heptane (price of industrial kerosene: 40 Japanese yen/L; industrial n-heptane: 200 Japanese yen/L).

3.2 Effect of kerosene concentration and soaking time on toner softening

The degree to which toner was softened in kerosene was measured with a tackiness tester to investigate the correlation of toner resin softening versus soaking time. Changes in the degree of softening (pulling stress) with soaking time when tablet toner samples were soaked in undiluted kerosene, 1% kerosene, and 0.1% kerosene are shown in Figs. 5(a) and (b). The results when surfactants were added are also shown in these figures. When tablet samples were soaked in undiluted kerosene, the degrees to which both magnetic and non-magnetic toner softened rapidly increased during the first 10–15 min, and saturated in about 30 min. In 1% kerosene with no surfactant, the degree of softening also saturated in about 30 min, at about 70% of that for undiluted kerosene. Although the degrees to which both the magnetic and non-magnetic toner samples were softened also saturated in about 30 min in 0.1% kerosene, it decreased to about 20% of that for the case of undiluted kerosene. The addition of surfactants significantly lowered the degree of softening, which can be seen by comparing the results for emulsions having the same concentrations. The degree of softening for 1% kerosene containing surfactants was less than 10% of that for undiluted kerosene. For 0.1% kerosene containing surfactants, it did not change between pre- and post soaking. When an emulsion showed a stable dispersion state by the addition of a surfactant, the probability of kerosene coming into contact with a sample tablet was expected to increase along with the increase in surface area of the kerosene droplets. Although surfactants were added based on this expectation as mentioned in the previous paragraph, the results in Fig. 5 reveal that softening was hindered on the contrary. The cause of this result is regarded to be the sample tablets' decrease in adhesion probability by the kerosene droplets developing hydrophilic properties with the addition of the surfactants, and that the inhibition of contact of kerosene and sample tablets made the progression of the softening reaction more difficult. The degree of softening gradually decreased after reaching the maximum value. Since the change in the degree of softening in this experiment was measured by touching the same part of a sample tablet several times with a probe, the softened part was pushed away very slightly to the periphery of the probe in each test, especially when the degree of softening was high. Because this tendency was seen prominently after the degree of softening reached its maximum value, it is considered that the degree of softening had decreased. From the results of the softening tests, it is suggested that undiluted kerosene is the most effective solvent for toner liberation. It is also possible for 1% kerosene emulsion to achieve high toner liberation when dispersed with mechanical stirring without any addition of a surfactant. The optimum soaking time is confirmed to be 30 min.

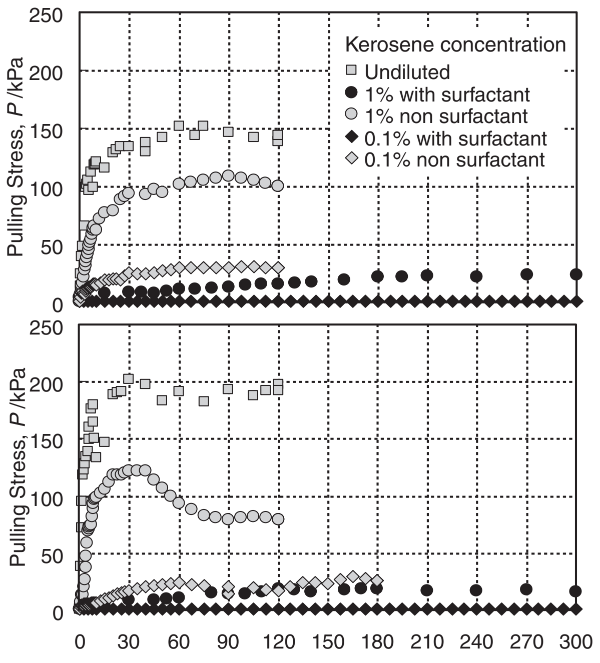

Fig. 5 Relation between soaking time and pulling stress (softening degree) of toner samples with some kerosene reagents. (a) Magnetic toner sample (b) Non-magnetic toner sample.

Conversely, adhesive materials in waste paper such as, for example, "stickies" or 3M Post-It Notes® caused trouble such as filter clogging and sticking with regenerated paper.[3,19,21] Although there is no clear definition of the adhesion border that caused the trouble, our previous studies show that the pulling stress of an acryl adhesive is about 100–120 kPa.[22] The stress of softened toner in this study showed the same or higher value than that of an acryl adhesive, but the stress rapidly decreased to less than 20 kPa after transference into water or sodium hydroxide solution.

3.3 Effect of disintegration process on toner softening and liberation

In the conventional deinking process that is used for conventional waste paper, paper disintegration and ink liberation are carried out easily because paper swelling and the saponification of oil-based ink occur simultaneously in sodium hydroxide solution.[23] However, as paper does not swell when soaked in kerosene, the order in which it is soaked in alkali (sodium hydroxide solution) for swelling and kerosene for toner softening is assumed to affect the degree of toner liberation. In this paragraph, toner states were compared in the case of carrying out alkali soaking before and after kerosene soaking. Results of the treatments when kerosene soaking was followed by alkali soaking and then washing with water are shown in Figs. 6(a) and (b). The results when alkali soaking was followed by kerosene soaking and then washing with water are shown in Figs. 7(a) and (b). When kerosene soaking was carried out first, little change could be seen in the magnetic toner (Fig. 6(a)).

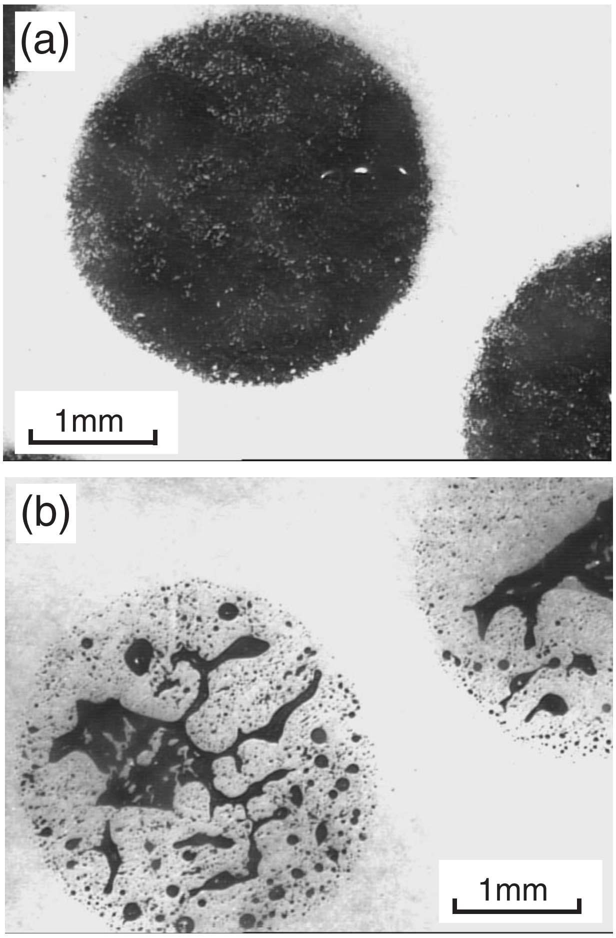

Fig. 6 Microscopic images of printed toner (treatment order: kerosene → sodium hydroxide solution). (a) Magnetic toner sample (b) Non-magnetic toner sample.

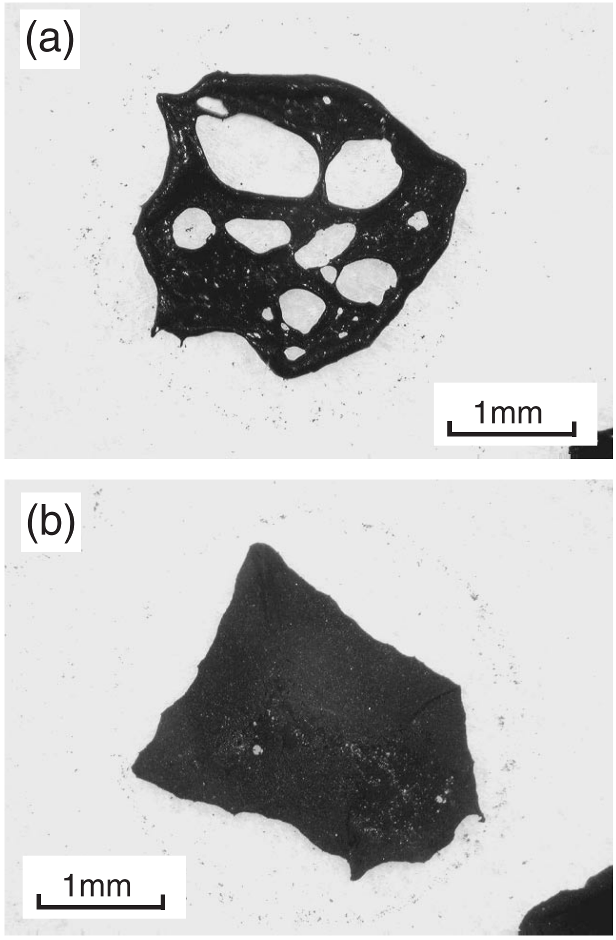

Fig. 7 Microscopic images of printed toner (treatment order: sodium hydroxide solution → kerosene). (a) Magnetic toner sample (b) Non-magnetic toner sample.

Although some non-magnetic toner particles were peeled off the paper, parts wedged in the paper fiber remained (black dots in Fig. 6(b)). This result suggests that the toner was softened but the wedged parts were not released by kerosene soaking, which means that the degree of softening in kerosene soaking cannot extend into the alkali soaking stage as mentioned above. It is considered that non-magnetic toner, for which a relatively high degree of softening was achieved, is cut away from the paper with the parts that remained wedged in the paper fibers, such as in Fig. 4(c), and that with magnetic toner, which showed a relatively low degree of softening, the original form is maintained without such cutting. When alkali soaking was carried out first (Fig. 7(a), (b)), no wedged parts remained in either case because the bound fiber loosens slightly from the swollen paper. This state lasts into the kerosene softening stage. Smooth exfoliation from the fringe of the circle followed by contraction of the softened toner was also observed in this experiment. This is considered to be a phenomenon such as hydrophobic aggregation of the hydrophobic softened toner (contact angle: 80–100°) trying to decrease its contact area to water. From the above results, it was confirmed that treatment carried out in the order of alkali soaking followed by kerosene soaking and then washing with water is effective for toner liberation.

3.4 Toner liberation by kerosene soaking

Toner liberation for samples printed with magnetic toner is easily measured using a microscope because all toner can be recovered by a magnet. So, liberation of the toner samples that had been disintegrated by the methods described in the previous section was investigated. The degrees of liberation for toner soaked with undiluted kerosene and kerosene emulsions are shown in Fig. 8. When only alkali soaking was carried out, as done in the conventional deinking process (A in Fig. 8), the degree of toner liberation was no more than 3.8% under weak agitation conditions (*i.e.*, 8.3 s^{-1} for 5 min). This indicates that toner could not be released by swelling the paper by alkali soaking, and that toner which has not been softened cannot even be cut from the wedged parts at the border, causing most of the toner to maintain its bind with the fiber (shown in Fig. 9(a)). Although one might think that the degree of liberation may be increased slightly by strongly agitating the sample within the range that fiber will not be cut, liberation at the interface of paper fiber and toner cannot be expected because only comminution of such particles shown in Fig. 9(a) would occur. The degree of toner liberation increased to 98.1% when samples were soaked in undiluted kerosene (A-100K) after alkali soaking, which means that almost perfect liberation was achieved. An 80.1% degree of liberation was obtained even for emulsions of

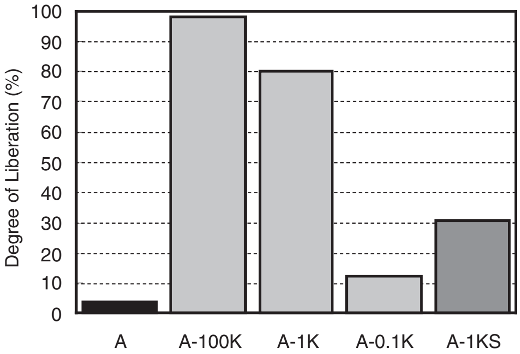

Fig. 8 Degree of liberation for magnetic toner under some disintegration conditions. A: Alkali soaking only (conventional method) A-100K: Alkali soaking + undiluted kerosene soaking* A-1K: Alkali soaking + 1%-kerosene soaking* A-0.1K: Alkali soaking + 0.1%-kerosene soaking* A-1KS: Alkali soaking + 1%-kerosene soaking with surfactant** *Kerosene soaking time: 30 min. **Kerosene soaking time: 180 min.

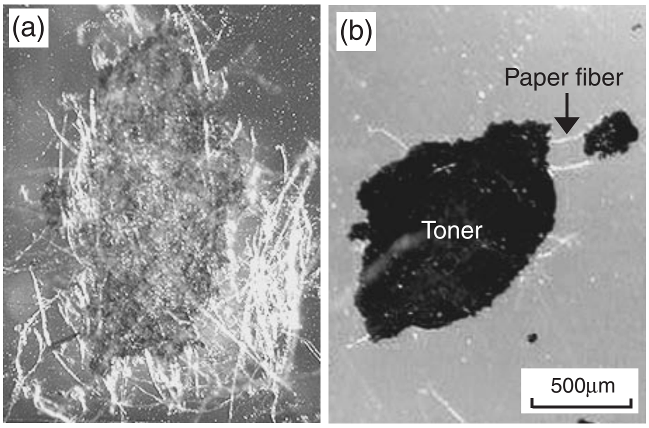

Fig. 9 Microscopic images of non-liberated toner particles in disintegrated model waste paper sample (a) disintegrated only by alkali soaking (b) disintegrated by 1%-kerosene soaking.

kerosene concentration lowered to 1% (A-1K), which would reach 100% if toner particles connected to only one or a few particles were included, such as in Fig. 9(b). But when the emulsion concentration was lowered to 0.1% (A-0.1K), the degree of liberation decreased to 12.5%. The reason why the degree of liberation is almost the same even when the concentration is diluted to 1/100 kerosene may be because kerosene droplets adhere selectively to hydrophobic toner. However, for 1/1000-diluted kerosene, the surface area of kerosene droplets decreased, which was why non-reacted toner was observed. By the addition of a surfactant to 1% kerosene emulsion to stabilize the kerosene dispersion (A-1KS), the degree of liberation decreased to 30.8% even when the soaking time was extended to 180 min. As previously mentioned in the section on the decrease in the degree of softening, this result is considered to have emerged because the surfactant lowered the adhesion probability of kerosene droplets to the toner as well as inhibiting the softening reaction even after adhesion.

Figures 10(a)–(e) show the size distribution in cumulative area of both the total amount of toner and liberated toner particles, which were calculated from the Heywood diameter.

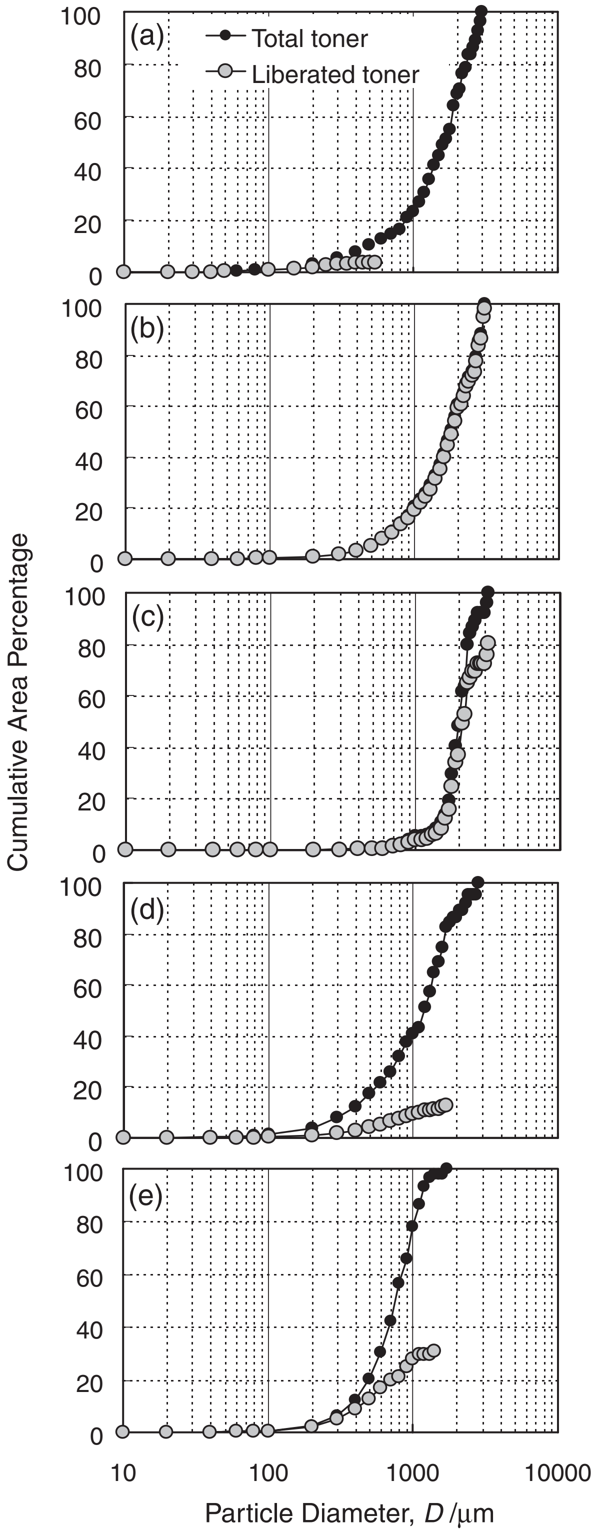

Fig. 10 Size distribution of total and liberated toner (a) A: Alkali soaking only (conventional method) (b) A-100K: Alkali soaking + undiluted kerosene soaking* (c) A-1K: Alkali soaking + 1%-kerosene soaking* (d) A-0.1K: Alkali soaking + 0.1%-kerosene soaking* (e) A-1KS:Alkali soaking + 1%-kerosene soaking with surfactant** *Kerosene soaking time: 30 min **Kerosene soaking time: 180 min.

When soaked with only alkali (Fig. 10(a)), only tiny particles of toner less than 600 μm in size were liberated, while those particles larger than 600 μm were not liberated at all, as shown in Fig. 9(a). On the other hand, when soaked in kerosene, the effect of the exfoliation of toner from paper by softening began to emerge and contributed to the liberation of toner particles over 1 mm in size. The diameter of liberated toner particles increased with increases in kerosene concentration (Figs. 10(b)–(d)). Toner particles of all sizes could be liberated in undiluted kerosene (Fig. 10(b)). Only a few toner particles over 2 mm were not liberated in 1% kerosene (Fig. 10(c)), but these particles appeared in the forms shown in Fig. 9(b). In 0.1% kerosene, toner over 2 mm in size could not be liberated at all; only particles of toner less than 2 mm in size were liberated (Fig. 10(d)). Because not only the dispersion of kerosene but also that of toner improved with the addition of a surfactant, the size distribution of all toner particles shifted to the range of smaller sizes (Fig. 10(e)). Although the maximum size of liberated toner particles was the same as the maximum size of all the toner particles, which was similar to the case without adding a surfactant (Fig. 10(c)), only part of the toner was liberated throughout all particle sizes in this case. Detailed analysis for samples printed with non-magnetic toner was not carried out because measuring toner liberation for them would have been difficult. But, from the results of the microscopic observations it can be presumed that almost the same degree of liberation as that for samples using magnetic toner could be achieved through the same kind of toner softening as mentioned above.

It has become apparent from these results that toner liberation can be realized with weak agitation at ambient temperature by soaking waste office paper in kerosene emulsions of concentrations over 1% for 30 min after soaking with sodium hydroxide solution. Two kinds of representative toner were used as samples in this study; however, strictly speaking, toner can be classified into numerous kinds by the content of resin and trace components. Therefore, liberation effects for many kinds of toner will have to be investigated in order to put this method to practical use.

4. Conclusion

In order to improve the degree of liberation between toner and paper, a disintegration method for waste office paper using kerosene was investigated in this paper. The results are summarized as follows.

(1) Each of the four kinds of solvent used in this study softened toner well, but the use of aromatic solvents such as toluene and xylene dissolved toner resin and thereby made the emulsion turn muddy gray in color. This gray color indicates the dispersion of fine pigments in the toner into the emulsion, and this phenomenon may result in a decrease of paper whiteness. Although the softening states were the same among kerosene and n-heptane emulsions, kerosene was selected as the most suitable softening reagent from the aspect of price.

(2) Softening tests of toner that had been softened with different concentrations of kerosene indicated that softening degrees were higher when treated in undiluted or 1% kerosene emulsions. The optimum soaking time was determined to be 30 min from the time required to reach the maximum softening degree. By adding a surfactant when making kerosene emulsions, the softening degree decreased significantly.

(3) From investigations into the order in which soaking in sodium hydroxide and kerosene were carried out, it was found that the wedged parts of toner that had infiltrated the paper tended to remain after the treatments when kerosene soaking was carried out first. It is important to loosen the fibers in advance by soaking them in sodium hydroxide solution in order to detach the toner, including the wedged parts. When kerosene soaking was carried out after alkali soaking and then washed with water, the softened toner exfoliated easily through contraction.

(4) The degree of toner liberation increased slightly to 12.5% from soaking with a 0.1% kerosene emulsion, and to 30.8% with a 1% kerosene emulsion when a surfactant was added. Under conventional alkali soaking, the degree of liberation was only 3.8%. The degree of liberation increased to 98.1% and 80.1% respectively when soaked in undiluted and 1% kerosene emulsion. Even the remaining unliberated toner particles were connected to one or a few fibers, and the degrees of liberation for both cases reached 100% if these kinds of toner particles were counted together.

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