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ORIGINAL PAPER

Recovery of valuable metal concentrate from waste printed circuit boards by a physical beneficiation technology

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Abstract It is greatly significant to separate precious metals from the waste printed circuit boards (PCBs) with appropriate methods for the resource recycling and environment protection. A combined physical beneficiation technology for the recovery of waste PCBs was investigated. Waste PCBs were disassembled into substrates and slots firstly. Waste PCB substrates were crushed to the size below 1 mm by a wet impact crushing approach. The double Rosin–Rammler functions were proposed to describe the particle size distribution characteristic of the substrates. The crushing products of the substrates were separated by a self-designed water-medium tapered column separation bed. The results indicate that the separation efficiency of 93.9 % and metal recovery rate of 93.7 % are obtained with a water discharge of 5.5 m^3/h, feeding capacity of 250 g/min and inclination angle of 35°. Waste PCB slots were crushed to the size range of 0.5–5 mm by a dry impact crushing approach and separated by an active pulsing air classifier. The separation results show that a separation efficiency of 92.4 % and metal recovery rate of 96.2 % are obtained with the airflow velocity of 2.90 m/s and pulsing frequency of 2.33 Hz. The mismatching components in the metal concentrate are relatively less with suitable operating conditions. Precious metals could be obtained by the further separation and purification of metal concentrates. The technology has great potential to be applied in the field of waste PCB recycling.

Keywords Waste printed circuit boards (PCBs) · Wet impact crushing · Water-medium tapered column separation bed · Active pulsing air classifier · Beneficiation flowsheet

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Introduction

More and more electronic wastes are generated around the world with the continuous development of electronic industry. Recycling and reutilizing of electronic wastes have been recognized as a great challenge for human beings; especially, the high-efficiency recycling of waste printed circuit boards (PCBs) is an important topic in the field of environment protection and resource recycling. Waste PCBs contain metallic elements such as Cu, Pb, Sn, Ni, Fe, Al, Cd, Be, and Pd, including the precious metals Ag and Au. A number of toxic and hazardous metal components, such as Pb, Cd, and Pd, exist in the waste PCBs with multiple existing states. These heavy metals may cause serious environment pollution. Meanwhile, waste PCBs are rich in numerous valuable constituents for recycling. Therefore, separating the hazardous components and recycling valuable constituents from waste PCBs are significantly important.

A number of researches on the recycling of waste PCBs have been investigated in recent years (Huang et al. 2009; Canal Marques et al. 2013; Birloaga et al.2013; Zhou and Qiu 2010; Xiu and Zhang 2010; Guo et al. 2011; Park and Fray 2009; Yang et al. 2012; Kara 2011; Das et al. 2009; Hall and Williams 2007; Veit et al. 2005; Eswaraiah et al. 2008; Duan et al. 2009; Zhan and Xu 2009; 2011; Wu et al. 2008). The common methods include dismantling, shredding, air separation electrostatic separation, triboelectrostatic separation, hydrometallurgy, and pyrometallurgy. However, the methods of dismantling and shredding could only achieve a primary treatment of waste PCBs. The method of traditional air separation shows lower separation efficiency for waste PCBs. The technology of electrostatic and triboelectrostatic separations has the disadvantages of limited treating capacity, which are difficult to be widely popularized in industry. The processes of hydrometallurgy and pyrometallurgy may create air and water pollutional problems, which have adverse effects on the environmental protection. The mechanical approaches are geared toward less environmental pollution, lower cost, higher material recovery, and easier industrialization. The mechanical recycling processes of PCBs can achieve full constituent recovery including ferrous metals, nonferrous metals, precious metals, and organic substances in PCBs. Therefore, the mechanical recycling processes gradually dominate the reutilization of waste PCBs (Murugan et al. 2008).

In this work, a fundamental study has been carried out by a mechanical recycling approach. A physical beneficiation technology was developed to recover the valuable metal concentrate from waste PCBs. The size-classification method of wet impact crushing has been applied to control the pollution caused by fugitive dust and odor. A water-medium tapered column separation bed and an active pulsing air classifier were utilized to achieve an effective classification of materials with different particle size distribution. The experimental results indicate that the metal concentrates could be recovered and recycled efficiently by the effective physical beneficiation flowsheet. Therefore, the physical beneficiation technology may not only achieve the pretreatment of waste PCBs (e.g., crushing and dissociation), but also could realize the high-efficiency separations of different-sized crushing products (e.g., subtracts and slots). The coarse-sized and fine-sized materials could be classified with different separation devices in the combined beneficiation flowsheet, which is greatly beneficial to increase the treating capacity and improve the separation efficiency. Additionally, the approaches of wet impact crushing and water-based separation are utilized to deal with the fine particles of waste PCBs. Thus, it avoids serious pollution problems. Overall, the physical beneficiation technology shows the advantages of large treating capacity, low treating cost, high separation efficiency, and little environmental pollution. It has great application potential to be developed and popularized in the field of waste PCBs processing. The achievements of this work could provide theoretical basis and useful guidance for the further development and application of the technology in the fields of environmental protection and waste recycling. The whole theoretical and experimental research was carried out from September 2005 to June 2013 in the laboratory of Mineral Processing in China University of Mining and Technology.

Materials and methods

Sample preparation

The waste PCBs used in this study were collected from an electric-waste recycling plant in Xuzhou, China. Initially, the waste PCBs were dismantled into the substrates and slots. Then, the waste PCB substrates and slots of different shapes and sizes were crushed into smaller particle sizes with different crushing methods, respectively. Subsequently, a self-designed double toothed roll crusher was used to crush waste PCBs into 20×20 mm by both compression and shearing actions in the primary shredding.

The waste PCB substrates mainly consist of copper-clad laminate, which should be sufficiently crushed in order to guarantee the complete dissociation of copper and other disseminated metals. Therefore, in the second-stage crushing, a wet impact crusher with the water medium was employed to achieve liberation of metals from other components contained in waste PCB substrates. Its schematic diagram is shown in Fig. 1. The blades are attached to rotating arms in a way that allows the blades to swing freely. As the arms rotate inside the drum, the swinging blades contact the feed material at a high speed. This imparts kinetic energy from the blades to the feed, fracturing the feed in the process. High-speed feed particles also fracture when they contact other particles or stationary parts of the crusher. Feed that escapes fracture after one impact is hit by the blades again. In this crusher, water is fed into an inlet. This causes the particles to form slurry that carries the broken particles through the sieve plate.

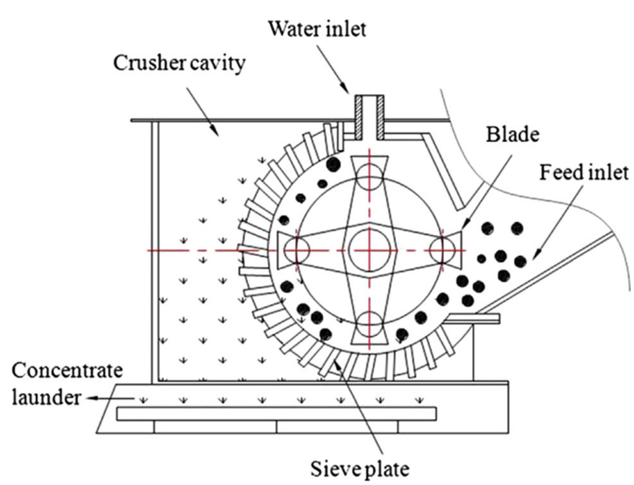

Fig. 1 Schematic diagram of wet impact crusher

Separating equipments

Based on computational fluid dynamic (CFD) and the separation theory of inclined flow, a water-medium tapered column separation bed was proposed and designed as a new separating equipment for recycling the crushing products of waste PCB substrates (Zhao et al. 2012). The experimental tapered column separation bed has a diameter of 200 mm on the top, a diameter of 120 mm in the bottom, and the total height of 800 mm. The water distributor is designed and installed at the bottom of separation bed so as to improve the stability and uniformity of water flow. The schematic diagram is shown in Fig. 2a.

The waste PCB slots were crushed into smaller particles by a normal dry impact crusher. Thus, a novel dry air classifier was proposed to carry out the separation experiments of waste PCB slots. The laboratory scale system of active pulsing air classifier is shown in Fig. 2b. The separation column with several segments is installed, which includes a group of organics glass columns of inner diameter 50 mm and length 250 mm being connected by flanges. The height of the separation column can be adjusted in the light of actual needs. Commonly, a 1,750 mm high column could meet the actual material separation. The active pulsing airflow is produced by a pulsing air generator, which includes pulsing valve, electromotor, and transducer. The frequency of the pulsing air can be adjusted with the transducer. The screw feeder controls the feeding forcibly and equally prevents the escape of dust as well. Different kinds of materials are separated into heavy particles and light particles by the difference of densities. The dust could be collected with a dust collector cyclone.

Results and discussion

Particle size distribution of the waste PCB crushing products

Particle size distribution model of the crushing waste PCB substrates

While using slippery hammerhead, the rotate speed of 882r/min, flux of water 5 m^3/h, and aperture of the sieve plate 2.2 mm, the results of wet impact crushing indicate that the metals degree of dissociation of waste PCB substrates could achieve above 95 % with the particle size decreasing to −1 mm. The test results of crushing products of waste PCB substrates are shown in Table 1. It could be obtained that 96.79 % of the crushing products distribute below 2 mm with the metal grade of 97.18 %. Therefore,

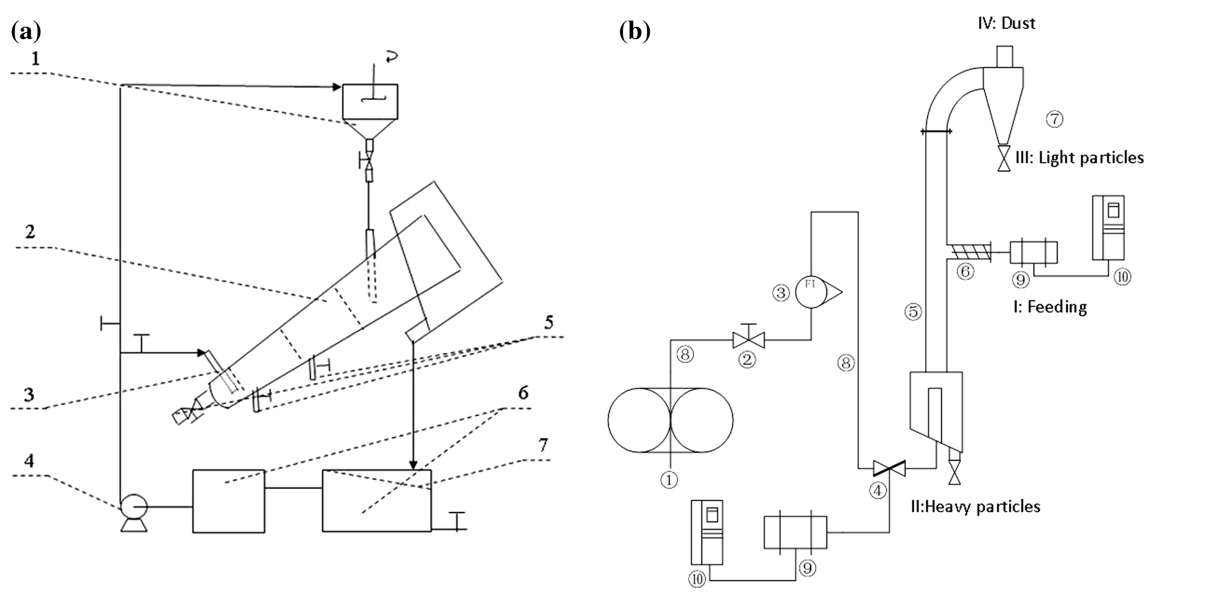

Fig. 2 Schematic diagrams: **a** the water-medium tapered column separation bed (*1* beater; *2* separation bed; *3* water distributor; *4* water pump; *5* discharge outlet; *6* circulation water tank; *7* filter cloth), **b** the laboratory scale system of active pulsing air classifier (① air blast; ② flow valve; ③ rotor flow meter; ④ pulsing valve; ⑤ separation column; ⑥ screw feeder; ⑦ cyclone dust collector; ⑧ pipe line; ⑨ electromotor; ⑩ transducer)

Table 1 Test results of waste PCB substrates by the wet impact crushing

Size (mm)	Production rate		Degree of liberation (%)	Metal grade (%)	Distribution rate (%)
	Weight ratio (%)	Positive accumulation yield (%)			
−2.2 + 2	3.21	3.21	66.00	22.75	2.82
−2 + 1	11.25	14.46	70.00	44.78	19.45
−1 + 0.5	19.01	33.47	95.00	53.96	39.61
−0.5 + 0.25	13.9	47.37	100.00	38.23	20.52
−0.25 + 0.125	10.88	58.25	100.00	21.05	8.84
−0.125 + 0.074	5.54	63.79	100.00		
−0.074	36.21	3.21	100.00	6.28	8.78
Total	100.00	–	–	26.51	100.00

the products with a particle size range below 1 mm were taken as the raw feeding material in the separation experiments.

The test results in Table 1 also indicate that the particle size distribution of crushing products of the waste PCB substrates presents the characteristic of bimodal distribution. It means that 1–0.5 and –0.074 mm are the dominant particle size ranges of the crushing products. The most common particle size characteristic functions in the crushing are Gates–Gaudin–Schutzman characteristic function, Rosin–Rammler characteristic function, and Lognormal distribution function, respectively. Based on the characteristics of above three particle size distribution functions, it could bring certain errors to describe the particle size distribution of the crushing products of waste PCB substrates whether applying any abovementioned function. The Rosin–Rammler particle size characteristic function is applicable to many types of crushers, ore mills, and classifiers. Thus, the previous researchers always employed the function to describe the particle size distribution of the crushing products. Finally, according to the bimodal distribution characteristic of the crushing products of waste PCB substrates, the double Rosin–Rammler particle size characteristic functions were proposed to represent the particle size distribution of the wet crushing products, which are also named DRR distribution functions, as shown in Eqs. 1 and 2.

$$W = \beta e^{-b_1 x^{n_1}} + (100 - \beta) e^{-b_2 x^{n_2}} \quad (1)$$

$$w = \beta n_1 b_1 x^{n_1 - 1} e^{-b_1 x^{n_1}} + (100 - \beta) n_2 b_2 x^{n_2 - 1} e^{-b_2 x^{n_2}} \quad (2)$$

where b_1, n_1, b_2, n_2, β refer to the coefficients, W refers to the positive accumulation yield of the crushing products with a particle size larger than x, w refers to the yield of each particle size range, x is the particle size of the crushing products.

The nonlinear fitting of the oversize accumulation yield was conducted with the self-defining DRR distribution functions. The fitting results are shown in Table 2. The evaluation indexes of the fitting results are correlation coefficient R^2 and residual error E, respectively. Normally, the closer R^2 value is to 1, the smaller the residual error E is, which indicates a more effective fitting function. It can be obtained from Table 2 that the correlation coefficient R^2 is much close to 1 with a smaller residual error E value of 0.1867. It indicates that the particle size distribution characteristic of the crushing products of waste PCB substrates could be correctly represented with the self-defining DRR distribution functions.

Particle size distribution of the crushing waste PCB slots

The monomer dissociation of waste PCB slots could be achieved in a larger size distribution, and the density differences of materials after impact crushing are comparatively large. So the crushing products can satisfy the experimental requirements of the raw materials size, density differences, and liberation degree. The test results of crushing products of waste PCB slots are shown in Table 3. The results indicate that the leading particle size of slots crushing products is 0.5–5 mm and takes up 92.84 % of the total products. Almost, all the metals distribute into the particle size range of 0.5–5 mm, accounting for 99.39 %. Therefore, 0.5–5 mm crushing products of waste PCB slots were selected as the raw feeding material in the separation experiments.

Table 2 Nonlinear fitting results of double Rosin–Rammler distribution function

Fitting parameters					Evaluation indexes	
b_1	n_1	b_2	n_2	β	E	R^2
1.43688	1.64941	2.34419	0.42326	34.6951	0.18671	0.99994

Table 3 Test results of waste PCB slots by the dry impact crushing

Size (mm)	Production rate		Degree of liberation (%)	Metal grade (%)	Distribution rate (%)
	Weight ratio (%)	Positive accumulation yield (%)			
−5 + 2	37.46	37.46	72.00	22.75	32.91
−2 + 1	34.77	72.23	85.00	44.78	49.80
−1 + 0.5	20.61	92.84	100.00	53.96	16.68
−0.5	7.16	100.00	100.00	38.23	0.61
Total	100.00	–	–	26.51	100.00

Table 4 Separation results of −1 mm waste PCB substrates

Water discharge (m^3/h)	Feeding capacity (g/min)	Inclination angle (°)	Concentrate grade (%)	Recovery rate (%)	Separation efficiency (%)
3.5	200	35	78.98	96.33	87.22
3.5	300	40	83.47	96.1	89.56
3.5	250	30	74.09	97.36	84.93
4.5	200	40	82.49	93.68	87.91
4.5	300	30	87.91	95.79	91.77
4.5	250	35	86.45	96.13	91.16
5.5	200	30	90.73	94.08	92.39
5.5	250	35	94.12	93.73	93.92
5.5	300	40	90.06	94.14	92.08

Separation experiments of waste PCB substrates

The water discharge, feeding capacity, and inclination angle are the major factors that affect the separation efficiency of the water-medium tapered column separation bed. The recovery rate, concentrate grade and separation efficiency are selected as the main indicators to evaluate the separation results (Raj et al. 2013; Uan et al. 2013; Uma Rani et al. 2013). The separation efficiency could be calculated as Eq. 3. The separation results of −1 mm PCB substrates are listed in Table 4. The experimental results show that the separation efficiency of higher than 85 %, recovery rate of more than 93 %, and concentrate grade of higher than 75 % could be achieved under certain suitable operating conditions.

$$\omega = \frac{(\alpha - \theta)(\beta - \alpha)}{\alpha(\beta - \theta)} \cdot 100\% \quad (3)$$

where ω refers to the separation efficiency, α,β,θ refer to the metal contents of feeding materials, metal concentrate, and nonmetallic concentrate, respectively.

The response surface methodology (RSM) approach was introduced to investigate the interactive relationships between the major factors and main indicators. It is widely applied to analyze the complex experimental results affected by multi-factors (Merrylin et al. 2014; Kavitha

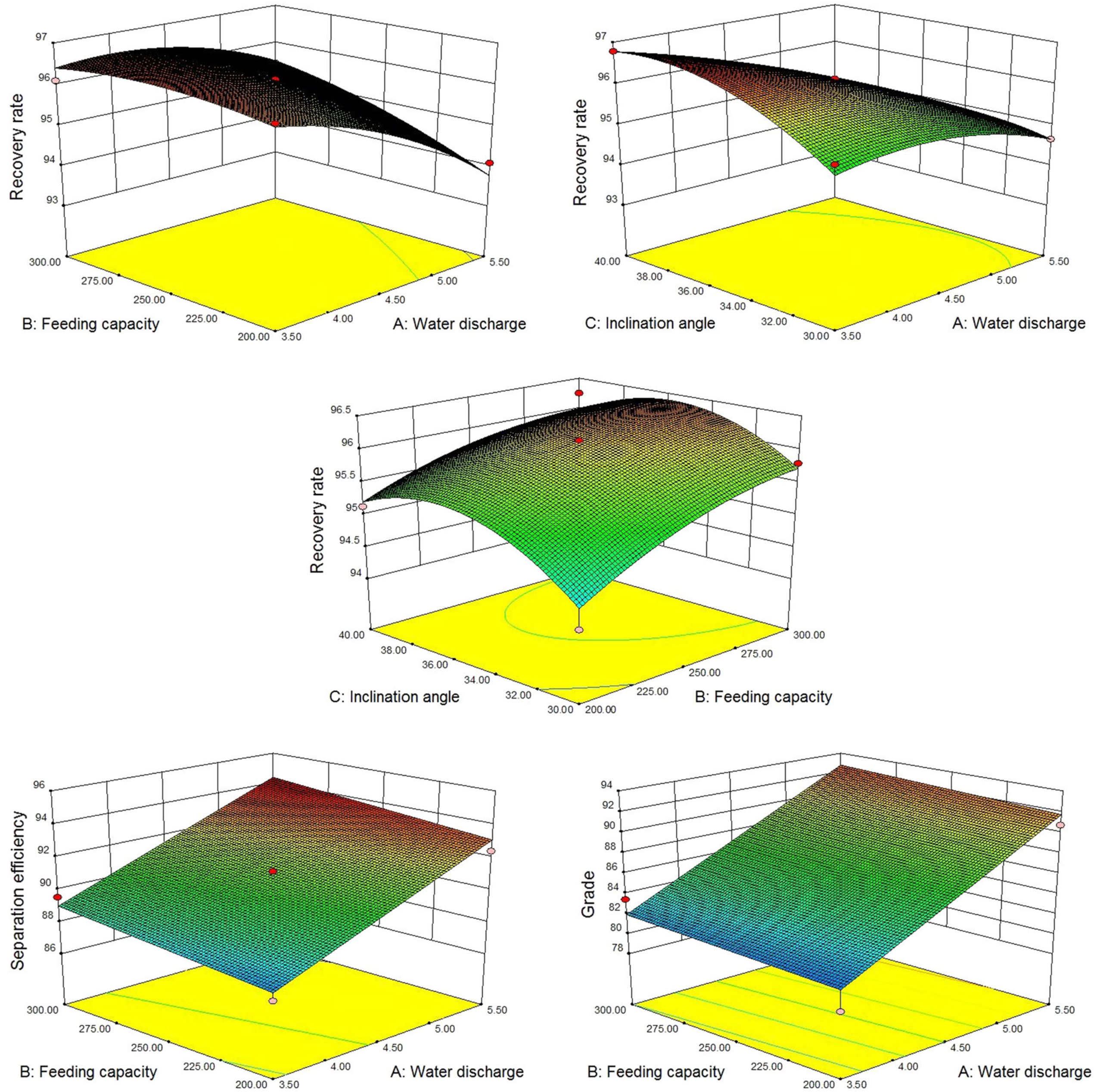

Fig. 3 The analyzing results with the response surface methodology (RSM) approach

et al. 2013; Kumar et al. 2012). The analyzing results with the RSM approach are shown in Fig. 3. It indicates that the recovery rate shows significant interactive relationships with the water discharge, feeding capacity, and inclination angle. Thus, in order to ensure the recovery rate of metal concentrate, it is better to maintain the water discharge of 3.5 m^3/h, the feeding capacity of 300 g/min, and the inclination angle of 35°. The results also indicate that the separation efficiency and concentrate grade show linear relationships with the three major factors. Normally, it means that the separation efficiency and concentrate grade gradually increase with the increasing of water discharge

Table 5 Separation results of 0.5–5 mm waste PCB slots

Airflow velocity (m/s)	Pulsing frequency (Hz)	Concentrate grade (%)	Recovery rate (%)	Separation efficiency (%)
2.76	1.96	76.30	96.16	89.66
2.76	2.14	79.39	95.53	90.25
2.76	2.33	80.87	95.98	91.27
2.90	1.96	82.41	95.31	90.56
2.90	2.14	83.35	95.22	91.13
2.90	2.33	84.62	96.16	92.43
3.04	1.96	82.43	94.19	90.22
3.04	2.14	82.77	94.40	91.74
3.04	2.33	83.86	95.44	92.26

and feeding capacity in most conditions. However, because of the capacity and structure limitations of the tapered column separation bed, the water discharge and feeding capacity should be maintained in a suitable variation extent. Furthermore, it could be seen from Table 4 that the recovery rate is almost more than 93 % with the water discharge of 3.5–5.5 m^3/h. It demonstrates that the factor of water discharge has less influence on the recovery rate of metal concentrate. Meanwhile, the results show that the water discharge has significant effect on the separation efficiency and concentrate grade. The separation efficiency is almost higher than 92 % with the concentrate grade of higher than 90 % with the water discharge of 5.5 m^3/h; especially, the separation efficiency of 93.92 %, the recovery rate of 93.73 %, and the concentrate grade of 94.12 % could be obtained with the water discharge of 5.5 m^3/h, feeding capacity of 250 g/min, and inclination angle of 35°. Compared with other physical separation methods, the distribution of particle size was broaden using tapered column separation bed with water medium to recover metals from PCBs, and lower limit size of separation almost approached to zero.

Separation experiments of waste PCB slots

Based on the separation mechanism of the active pulsing air classification and the results of basic experiments, airflow velocity, pulsing frequency, and feeding capacity are selected as the main factors influencing the separation efficiency (He et al. 2012; 2012). The related experimental results indicate that the influence caused by feeding capacity was relatively smaller than the other two factors. So, the airflow velocity and pulsing frequency were chosen as the leading factors. An orthogonal test with two factors and three levels was designed and conducted.

The separation results of 0.5–5 mm PCB slots are summarized in Table 5. It could be obtained that the highest separation efficiency of 92.43 % and metal recovery rate of 96.16 % could be obtained with the airflow velocity of 2.90 m/s and pulsing frequency of 2.33 Hz.

In order to further evaluate the separation effect, the content of mismatching components was introduced to analyze the influence of airflow velocity and pulsing frequency on the separation efficiency. The nonspecified component that mixed into the products is named mismatching component, which could be summarized as Eq. 4.

$$m_0 = m_\mathrm{h} + m_\mathrm{l} \tag{4}$$

where m_0 refers to total content of mismatching component, m_h refers to the component quantity mixed into the nonmetallic concentrate with a density larger than the separating density, m_l refers to component quantity mixed into the metal concentrate with a density smaller than the separating density.

The calculation quantity results of mismatching components of waste PCB slots are summarized in Table 6. It could be obtained that the mismatching content of metal concentrates reduces from 23.70 to 12.23 % with the airflow velocity increasing from 2.76 to 3.04 m/s and pulsing frequency increasing from 1.96 to 2.33 Hz. Meanwhile, the mismatching content of nonmetallic concentrates increases from 2.13 to 3.86 %. The results indicate that the quantity of nonmetallic component mixed into the metal concentrate obviously decreases with the increasing of airflow velocity and pulsing frequency. Oppositely, the quantity of metal component mixed into the nonmetallic concentrate is very less. It means that the airflow velocity and pulsing frequency should be increased within suitable extents so as to improve the product quality.

Beneficiation flowsheet based on a combined physical treating technology

Based on above two basic processes to deal with waste PCBs, an effective physical beneficiation technology to recover the metal concentrate from waste PCBs was proposed. The treating flowsheet based on the combined physical beneficiation technology is shown in Fig. 4. On the one hand, the optimal separation efficiency of 93.92 % and metal recovery rate of 93.73 % are achieved in the separation of waste PCB substrates. On the other hand, the optimum separation efficiency of 92.43 % and metal recovery rate of 96.16 % are achieved in the separation of waste PCB slots. The overall treatment of waste PCBs could be simultaneously completed by different physical separation technologies in the same beneficiation flowsheet.

The experimental results were also compared with the achievements by other beneficiation technologies. For instance, a concentrate grade of over 93 % total metal at a recovery of over 54 % or a grade of 66 % total metal at 95 % recovery could be achieved by a combination of dry and wet treating processes (Das et al. 2009). Metallic elements in waste PCBs were concentrated effectively by the approach of supercritical methanol treatment with the content of metals up to 62 % under optimum conditions

Table 6 Calculation quantity results of mismatching components of waste PCB slots

Airflow velocity (m/s)	Pulsing frequency (Hz)	Quantity of nonmetallic concentrate m_l (g)	Quantity of metal concentrate m_h (g)	Mismatching component in nonmetallic concentrate (g)	Mismatching component in metal concentrate (g)	Mismatching content of metal concentrate (%)	Mismatching content of nonmetallic concentrate (%)
2.76	1.96	131.30	92.00	2.80	70.20	23.70	2.13
2.76	2.14	199.10	132.00	4.50	104.80	20.61	2.26
2.76	2.33	122.00	82.60	2.80	66.80	19.13	2.30
2.90	1.96	120.80	88.70	3.60	73.10	17.59	2.98
2.90	2.14	158.00	114.70	4.80	95.60	16.65	3.04
2.90	2.33	132.60	92.30	4.50	78.10	15.38	3.39
3.04	1.96	133.60	90.50	4.60	77.20	14.70	3.44
3.04	2.14	149.10	99.80	5.40	86.50	13.33	3.62
3.04	2.33	142.50	97.30	5.50	85.40	12.23	3.86

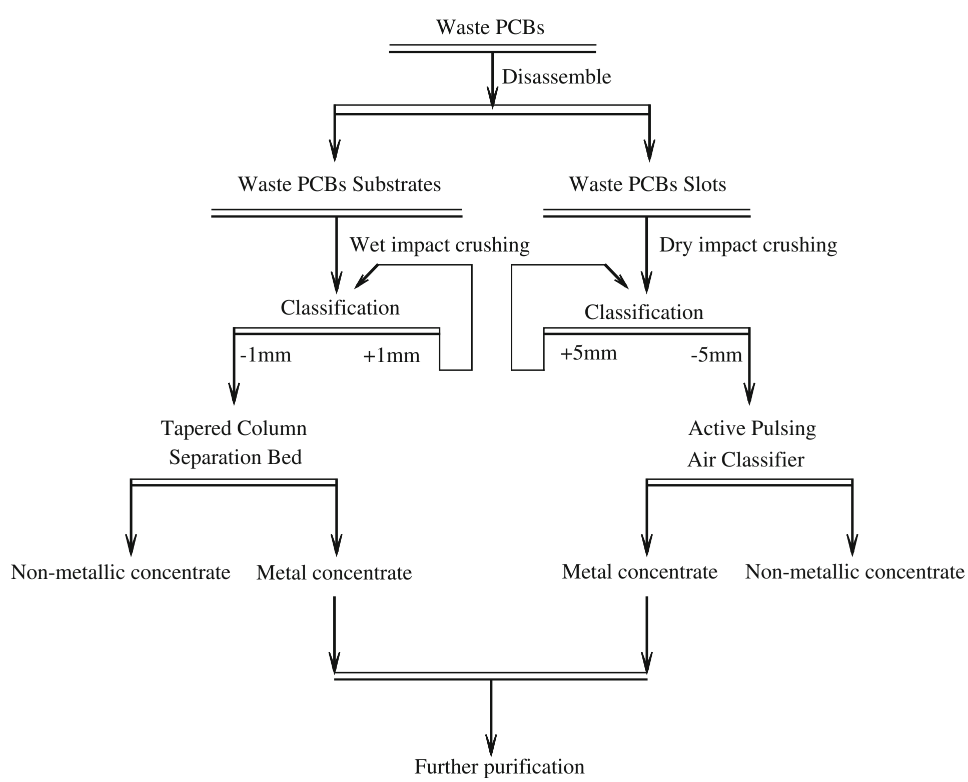

Fig. 4 The beneficiation flowsheet based on a combined physical treating technology of waste PCBs

(Xiu and Zhang 2010). The approach of froth flotation was utilized for the beneficiation of PCB comminution fines. The experimental performance shows that an optimum combination of over 72 % recovery and over 55 % metal concentrated mass is obtained by the metallic enrichment of the sink (Ogunniyi and Vermaak 2009). Finally, the comparison results indicate that a higher separation efficiency and metal recovery rate of waste PCBs could be achieved by the beneficiation flowsheet in this studying. The precious metals could be obtained by the further separation and purification of metal concentrates.

Conclusion

In this investigation, a wet impact crusher was employed to achieve the comminution and liberation of metals from other components contained in waste PCB substrates. When using the slippery hammerhead, rotate speed of 882r/min, flux of water 5 m^3/h, and aperture of the sieve plate 2.2 mm, the metal dissociation degree of the waste PCB substrates achieves above 95 % with the particle size decreasing to −1 mm, indicating favorable dissociation. The double Rosin–Rammler functions were proposed to describe the particle size distribution characteristic of the substrates.

A self-designed water-medium tapered column separation bed was adopted to separate the crushing products of waste PCB substrates. The separation efficiency of 93.92 % and metal recovery rate of 93.73 % could be obtained with the water discharge of 5.5 m^3/h, feeding capacity of 250 g/min and inclination angle of 35°. An effective classification of the crushing products of waste PCB slots was realized with an active pulsing air classifier. The separation efficiency of 92.43 % and metal recovery rate of 96.16 % could be obtained with the airflow velocity of 2.90 m/s and pulsing frequency of 2.33 Hz. The mismatching content of metal concentrates reduces sharply with the increasing of airflow velocity and pulsing frequency within suitable variation ranges.

Based on the waste PCB substrates separation with a water-medium tapered column separation bed and waste PCB slots separation with an active pulsing air classifier, a combined physical beneficiation flowsheet was established to recover valuable metal concentrate from waste PCBs, which has great potential to be applied in the field of waste PCB recycling.

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