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Using vacuum pyrolysis and mechanical processing for recycling waste printed circuit boards

Laishou Long[a,b,*], Shuiyu Sun[a], Sheng Zhong[a], Wencan Dai[a], Jingyong Liu[a], Weifeng Song[a]

[a] Faculty of Environmental Science and Engineering, Guangdong University of Technology, 100 Waihuan Xi Road, Guangzhou Higher Education Mega Center, Guangzhou 510006, PR China
[b] Faculty of Chemistry and Environmental Engineering, Shaoguan University, Daxue Road, Shaoguan 512005, PR China

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ABSTRACT

The constant growth in generation of waste printed circuit boards (WPCB) poses a huge disposal problem because they consist of a heterogeneous mixture of organic and metallic chemicals as well as glass fiber. Also the presence of heavy metals, such as Pb and Cd turns this scrap into hazardous waste. Therefore, recycling of WPCB is an important subject not only from the recovery of valuable materials but also from the treatment of waste. The aim of this study was to present a recycling process without negative impact to the environment as an alternative for recycling WPCB. In this work, a process technology containing vacuum pyrolysis and mechanical processing was employed to recycle WPCB. At the first stage of this work, the WPCB was pyrolyzed under vacuum in a self-made batch pilot-scale fixed bed reactor to recycle organic resins contained in the WPCB. By vacuum pyrolysis the organic matter was decomposed to gases and liquids which could be used as fuels or chemical material resources, however, the inorganic WPCB matter was left unaltered as solid residues. At the second stage, the residues obtained at the first stage were investigated to separate and recover the copper through mechanical processing such as crushing, screening, and gravity separation. The copper grade of 99.50% with recovery of 99.86% based on the whole WPCB was obtained. And the glass fiber could be obtained by calcinations in a muffle furnace at 600 °C for 10 min. This study had demonstrated the feasibility of vacuum pyrolysis and mechanical processing for recycling WPCB.

1. Introduction

Printed circuit boards (PCB) are a major part of electrical and electronic equipment (EEE) and the waste printed circuit boards (WPCB) form about 3% by weight of the total amount of waste electrical and electronic equipment (WEEE) [1]. New technological innovation continues to accelerate the replacement of equipment leading to a significant increase of WPCB. The WPCB, which contain various valuable metals together with hazardous material, are thus considered both an attractive secondary resources and an environmental contaminant [2,3]. However, the WPCB are particularly problematic to recycle because of the heterogeneous mix of organic material, metals, and glass fiber [4]. This demonstrates the need to search for solutions of this kind of scrap, in order to have it disposed in a proper way, without harming the environment. Therefore, recycling of WPCB is an important subject not only from the recovery of valuable materials but also from the treatment of waste.

Many studies, such as pyrometallurgy [5], hydrometallurgy [6,7] and mechanical–physical process [8–13], have been carried out with regard to the recycling of WPCB. However, these existing processes of recycling WPCB generate atmospheric pollution through the release of dioxins and furans or high volumes of effluents [14]. Moreover, the current recycling methods only focus on the recovery of the valuable metals contained in the WPCB. Therefore, continued study on the recycling of WPCB is necessary.

Pyrolysis is a thermal recycling technique that has been widely researched as a method of recycling synthetic polymers [15–18] including polymers that are mixed with glass fibers [19]. Although a significant amount of research into the pyrolysis of WPCB has been reported, most of the work has been carried out under nitrogen atmosphere using analytical pyrolysis techniques or laboratory scale reactors [4,20–23]. Also, most of the work into the pyrolysis of WPCB has concentrated on the compositions of the organic products obtained [24–27], pyrolytic kinetics [24–26,28–30], and brominated compound formation and fate [22,24,27–29]. However, few studies have focused on vacuum pyrolysis and mechanical processing for recycling WPCB.

* Corresponding author at: Faculty of Environmental Science and Engineering, Guangdong University of Technology, 100 Waihuan Xi Road, Guangzhou Higher Education Mega Center, Guangzhou 510006, PR China.
E-mail address: laishou1013@hotmail.com (L. Long).

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Table 1
Components of WPCB.

Components	Content (wt.%)
Copper	37.5
Glass fiber	27.4
Organics	35.2

In this work, a process technology containing vacuum pyrolysis and mechanical processing was employed to recycle WPCB. At the first stage of this work, the WPCB was pyrolyzed under vacuum by using a self-made batch pilot-scale fixed bed reactor. At the second stage, the pyrolysis residues were crushed by using a high-speed shearing machine to liberate the copper, and then the crushed pyrolysis residues were classified into fractions of <0.105, 0.105–0.28, 0.28–0.45, 0.45–0.9, 0.9–2.0 and 2.0–4.0 mm. The fractions of classified pyrolysis residues were separated respectively into a light fraction of mostly non-metallic components and a heavy fraction of copper by gravity separation using a vertical zigzag airflow separator. The aim of this work was to develop a recycling process without negative impact to the environment to settle the problem of WPCB.

2. Materials and methods

2.1. Materials

The WPCB without electronic components used for the experiments originated from a local PCB factory. There was only copper coating on the epoxy base plates. And the WPCB was multi-layered structure, containing epoxy resin, woven glass fiber, brominated epoxy resin flame retardant and copper. The components of WPCB were given in Table 1.

2.2. Methods

The recycling process comprised vacuum pyrolysis and mechanical processing. At the first stage of this work, approximately 3 kg of WPCB were first cut into fragments of about 5 cm × 5 cm using an electrical saw and then pyrolyzed under vacuum in a fixed bed reactor to separate and recover organic resins. At the second stage, the residues obtained at the first stage were investigated to separate and recover the copper and glass fiber through mechanical processing, such as crushing, screening, as well as gravity separation. A schematic diagram investigated for recycling WPCB was shown in Fig. 1.

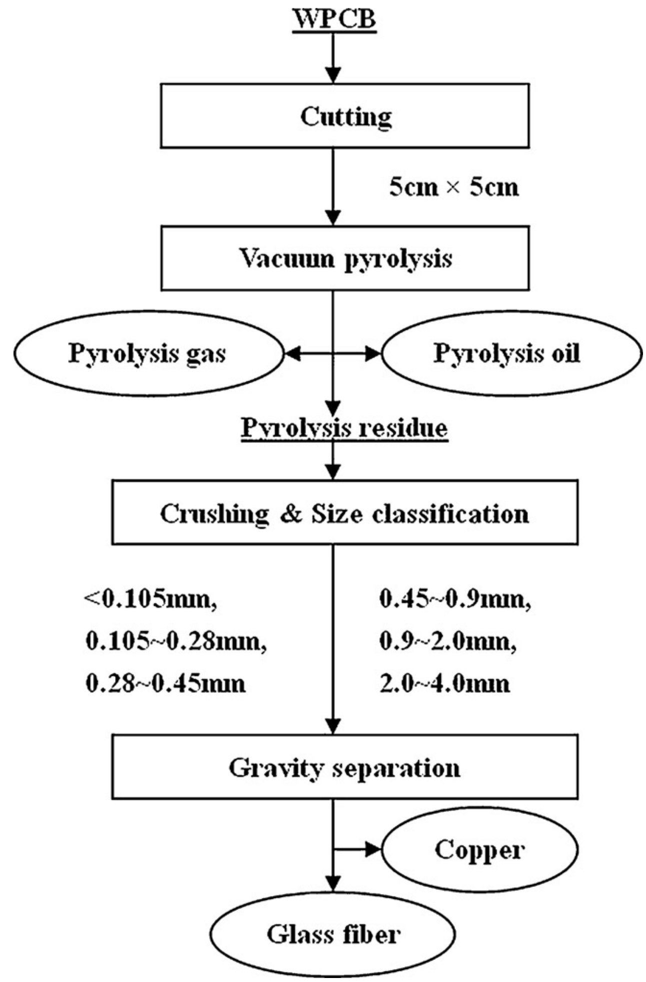

Fig. 1. Experimental scheme for recycling WPCB.

2.2.1. Vacuum pyrolysis

The vacuum pyrolysis experiments were carried out by using a self-made batch pilot-scale fixed bed reactor according to our previous research [31]. The reactor measured 350 mm in length by an internal diameter of 160 mm and was externally heated by a 10 kW electric heating furnace. Fig. 2 showed a schematic diagram of the vacuum pyrolysis apparatus, which included electric heating furnace, stainless steel fixed bed reactor, temperature control system, condensing system, vacuum system and gases treatment and collection system.

In a typical run, approximately 3 kg of WPCB pieces of about 5 cm × 5 cm were placed into the reactor at the start of the experiment, and the reactor was then sealed and vacuumed to the total pressure of 20 kPa by water ring vacuum pump before being heated to 550 °C at a rate of 10 °C/min. Once the reactor had reached 550 °C it was held at this temperature for 120 min to ensure that vacuum pyrolysis of the sample was complete, and then natural cooling to room temperature under vacuum. After exiting the fixed bed reactor, the pyrolysis gases and oils passed through a water-cooled condenser and then an ice-cooled condenser (ice salt water less than −5 °C) that collected any oils and waxes released during the vacuum pyrolysis process. In addition, a glass wool trap was used to remove any oils or waxes that were not trapped by the condensers. The water trunk contained 5 wt.% sodium hydroxide solution was used to collect water-soluble compounds in the exhaust gas. The organic and permanent gases were sampled by drawing off gas samples into a syringe in order to analyze by gas chromatography and collected using a gas bag. The pyrolysis residues and oils yields were determined in each experiment by weighing the amount of each obtained, while the pyrolysis gases yields were calculated by difference.

2.2.2. Crushing and size classification

In order to liberate the different components, especially to strip metal from the base plates, the vacuum pyrolysis residues of WPCB was crushed by using a high-speed universal crusher (FW-200, Tianjin Huaxin Instrument Factory, China). The shearing action generated by the rotor cutters and stator cutters crushed residues to small particles. The crushed materials coming out of the high-speed shearing machine were then classified into <0.105, 0.105–0.28, 0.28–0.45, 0.45–0.9, 0.9–2.0 and 2.0–4.0 mm by means of a vibrating screen with a standard sieve. The fractions of >4.0 mm were fed back into the crusher to crush again. The mass of these fractions was then determined by weighing, and the average copper content of these fractions were determined to investigate the distribution of copper for each particle size.

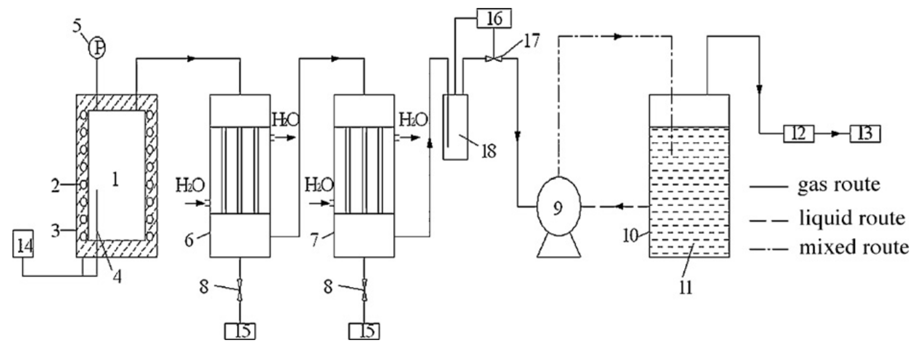

Fig. 2. Schematic diagram of the vacuum pyrolysis apparatus: (1) pyrolysis reactor, (2) heater, (3) temperature controlled furnace, (4) thermocouple, (5) vacuum meter, (6) water-cooled condenser, (7) ice-cooled condenser, (8) ball valve, (9) water ring vacuum pump, (10) water trunk, (11) sodium hydroxide solution, (12) active carbon, (13) gas bag, (14) temperature controller, (15) liquid collector, (16) gas sampling point, (17) valve, and (18) glass wool trap.

2.2.3. Gravity separation

Gravity separation is an important technique suitable for separating the samples with different density. The gravity separation experiment was carried out using a vertical zigzag airflow separator, as shown in Fig. 3. The gravity separation experiments proceeded under the different air velocities in the zigzag channel according to the particle sizes. The classified vacuum pyrolysis residues were separated into a light fraction of mostly non-metallic components and a heavy fraction of copper. As shown in Fig. 3, the bottom of separator collected copper particles and the bottom of hydrocyclone collected non-metal particles. The requisite air velocities were adjusted through tests for each particle size of the classified vacuum pyrolysis residues of WPCB. In this work, the air velocities in the zigzag channel needed for the various particle sizes were controlled as follows: 1.1 m/s for <0.105 mm, 1.3 m/s for 0.105–0.28 mm, 1.5 m/s for 0.28–0.45 mm, 1.8 m/s for 0.45–0.9 mm, 2.5 m/s for 0.9–2.0 mm and 3.3 m/s for 2.0–4.0 mm at a constant feed rate of 150 g/min. The average copper content in the heavy and light fractions were analyzed to assess the enrichment of the copper with the particle size of the classified vacuum pyrolysis residues.

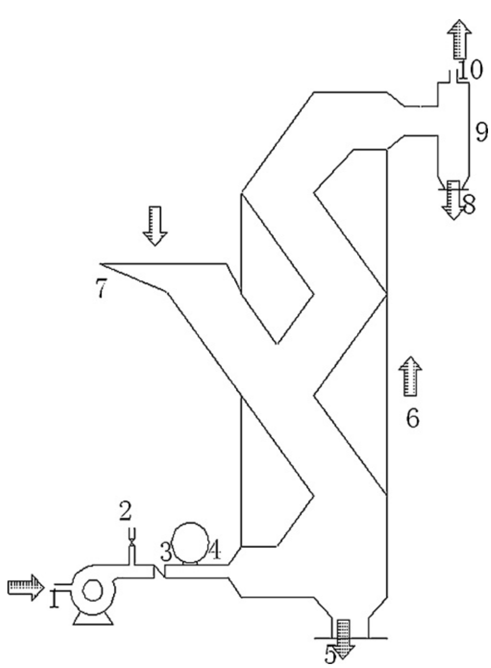

Fig. 3. Schematic diagram of the vertical zigzag airflow separator: (1) blower, (2) gate valve, (3) butterfly valve, (4) air flow meter, (5) heavy fraction, (6) updraft, (7) feed inlet, (8) light fraction, (9) hydrocyclone, and (10) air.

2.2.4. Analysis

The liberation degree of copper was determined by grain-counting following the method of Zhang and Forssberg [32]. Copper concentration was analyzed with an atomic absorption spectrometry (WFS-210, Beijing Rayleigh Analytical Instrument Corporation, China). The vacuum pyrolysis oils were analyzed with a GC-MSD (7890A/5975C, Agilent Technologies, USA). The organic and permanent gases were analyzed with a GC-FID and GC-ECD (GC-2014, Shimadzu, Japan). The amount of coke formed in the vacuum pyrolysis process and the unpyrolyzed organic matter were determined by subtraction method according to ignition loss test.

3. Results and discussion

All the fractions of the WPCB were separated and recovered through vacuum pyrolysis and mechanical processing. And the recycled products were respectively oils, copper and glass fiber. Schematic illustration of recycling WPCB was shown in Fig. 4.

3.1. Vacuum pyrolysis

The aim of vacuum pyrolysis was to separate and recover the organics from the WPCB without negative impact to the environment. The weight percentage of residues, oils and gases obtained in the WPCB vacuum pyrolysis experiments was 74.7%, 15.0% and 10.3%, respectively. The main composition of pyrolysis products were presented in Table 2.

3.1.1. Pyrolysis residues

The pyrolysis residues obtained by vacuum pyrolysis was shown in Fig. 4. The pyrolysis residues mainly consisted of glass fiber, copper and carbon. And the composition of the pyrolysis residues was presented in Table 2.

It could been seen from Fig. 4 that the residues obtained in the WPCB vacuum pyrolysis experiments was composed of pieces of equal dimensions to those of the original ones, and the color of WPCB after pyrolysis was changed to completely black, which was coke like carbonaceous product formed during the process due to cyclization and secondary repolymerization reactions [27,28]. Furthermore, the delamination between copper and other components appeared due to the results of complex physicochemical changes taking place in different material components during the vacuum pyrolysis processing [31]. From the XRD analysis of residues (Fig. 5), only SiO_2 and Cu were detected in the residues. However, copper oxides were not detected by XRD which may be attributed to the minor content of copper oxides. As the pyrolysis experiments were carried out under vacuum during the vacuum pyrolysis processing, little copper in the residue was oxidized to copper oxides. The

Fig. 4. Schematic illustration of recycling WPCB.

Table 2
Main composition of pyrolysis products resulting from the vacuum pyrolysis of WPCB.

Oils		Gases		Residues	
Components	Area%	Components	Area%	Components	wt.%
Acetone	2.68	H_2	5.1	Copper	50.6
Toluene	1.22	CO	28.6	Glass fiber	36.9
Benzofuran	1.20	CO_2	50.4	Carbon	12.5
2-Methyl-2,3-dihydro-benzofuran	2.17	CH_4	7.6		
2-Methylbenzofuran	1.86	C_2H_4	0.7		
Benzophenone	1.02	C_2H_6	1.5		
2,3-Dimethylbenzofuran	1.00	Propene	1.9		
2-Bromophenol	4.34	Propane	1.2		
Phenol	37.99	Butene	0.8		
2-Ethylphenol	1.46	Butane	0.3		
4-Methylphenol	5.66	HBr	0.8		
2-(1-Methylethyl)phenol	1.46	CH_3Br	1.1		
3-Ethylphenol	6.89				
Thymol	0.80				
4-(1-Methylethyl)phenol	23.81				
2,6-Dibromophenol	0.17				

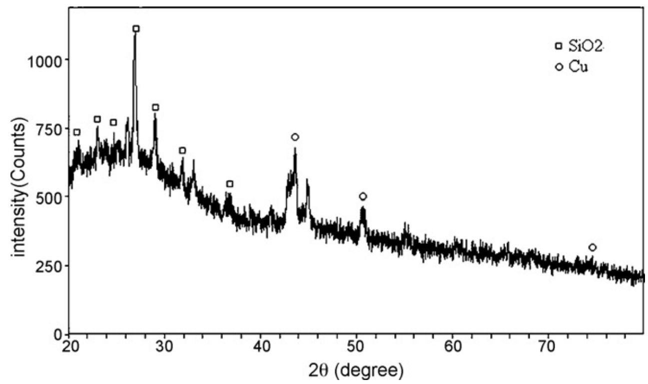

Fig. 5. XRD results of vacuum pyrolysis residues of WPCB.

copper was indicated to be simple substance of copper with high purity.

3.1.2. Pyrolysis oils

The WPCB pyrolysis liquids were a mixture of organic compounds, usually termed oils, with some aqueous products. Such liquids were centrifuged before their analyses in order to separate the aqueous phase, which was ~10 wt.%. The water could either be a product derived from the oxygen-containing functional groups during the process or due to the original moisture in the samples [33].

The major components of the oils obtained were identified and their corresponding % area was quantified by GC–MS. The compounds identified were presented in Table 2. As could be seen from Table 2, the oils were mainly composed of phenol, substituted phenols, benzofuran and substituted benzofurans. However, it was worth mentioning that the pyrolysis oils contained a significant number of brominated compounds, the most abundant of which was 2-bromophenol and 2,6-dibromophenol. Therefore, the oils could be used as fuels or chemical material resources after the proper treatment. The major components of the pyrolysis oils could all be attributed to the direct decomposition products of bisphenol A epoxy resin due to the cleavage of C–Br, C–C and O–CH_2 bonds. The most significant product in all the oils was phenol, followed by 4-(1-methylethyl) phenol, which must originate from the splitting of the bisphenol A structure [4]. However, the brominated compounds in the pyrolysis oils were most likely due to the presence of brominated epoxy resin in the WPCB [4,20].

3.1.3. Pyrolysis gases

The composition of the WPCB pyrolysis gases was shown in Table 2. It could be seen from Table 2 that by far the most abundant gases were carbon monoxide (CO) and carbon dioxide (CO_2), which presumably formed during the decomposition of the epoxy group but could also have been formed by the decomposition of calcium carbonate fillers in the polymer. The other significant components of the pyrolysis gases were methane and hydrogen, which might be derived from the cracking of methyl groups of the polymer in the WPCB. However, it was worth mentioning that the pyrolysis gases also contained some brominated compounds such as methyl bromide and hydrogen bromide. The small amounts of bromine that were present could easily be removed from combustion gases using existing technologies such as wet or dry scrubbing systems that were currently used in municipal waste incineration. The pyrolysis gases contain a significant proportion of CO_2 which would lower the calorific value of the gas. However, systems now exist for removing CO_2 from gas streams [4]. Therefore, the organic gases produced during pyrolysis process could be used as a fuel to provide the heat necessary for the pyrolysis of WPCB after the proper treatment.

3.2. Crushing and size classification

For the mechanical separation, liberation of values from non-values is the first step and crucial before separation can occur. The liberation of metals can be obtained by particle size reduction. In this work, the purpose of crushing was to strip copper from the base plates of vacuum pyrolysis residues of WPCB. In this experiment, the residues were crushed to liberate the copper by using a high-speed universal crusher and then classified according to particle size into six different fractions: <0.105, 0.105–0.28, 0.28–0.45, 0.45–0.9, 0.9–2.0 and 2.0–4.0 mm. The crushing results of liberation degree of copper, distribution of crushed products, copper grade, distribution of non-metallic components and distribution of copper with the particle size were shown in Table 3.

It could be seen from Table 3 that in the fractions of size <4 mm, the copper achieved approximately complete liberation by using a high-speed universal crusher due to lower interfacial bonds of the materials after vacuum pyrolysis treatment. Consequently, the high-speed universal crusher equipped with cutters using cutting stresses was effective for liberating the copper present in vacuum pyrolysis residue of WPCB.

As shown in Table 3, the crushed products were mostly distributed in the coarser fractions of size >0.45 mm and the finer fractions of size <0.28 mm, while relatively less in the intermediate fractions of size 0.28–0.45 mm. The distribution percentage of size >0.45, <0.28 and 0.28–0.45 was 53.95%, 37.03% and 9.02% respectively. Therefore, it could be inferred from the crushing results that the high-speed universal crusher could realize selective crushing of the vacuum pyrolysis residues of WPCB. Moreover, the grade of the copper became higher with increasing particle size. It increased from 0.04% to about 96% with increasing particle size from <0.105 to 2.0–4.0 mm.

It could be seen from Table 3 that the copper was concentrated in the coarser fractions of the crushed vacuum pyrolysis residue of WPCB, whereas the non-metallic components in the finer fractions. The amount of copper in the fractions of size >0.45 mm was about 99% based on the whole WPCB, while approximately 92% of the non-metallic components were concentrated in the finer fractions with particle size <0.45 mm. The non-metallic components, composed of brittle glass fiber and pyrolytic carbon, were crushed to a smaller size due to its brittleness, whereas the copper to a coarser

Table 3
Crushing results of vacuum pyrolysis residue of WPCB.

Particle size (mm)	Liberation degree of copper (%)	Distribution of crushed products (%)	Copper grade (%)	Distribution of non-metallic Components (%)	Distribution of copper (%)
<0.105	100	14.57	0.04	29.53	0.01
0.105–0.28	100	22.46	1.04	45.07	0.45
0.28–0.45	100	9.02	5.05	17.37	0.90
0.45–0.9	100	13.26	84.03	4.29	21.86
0.9–2.0	100	28.49	95.01	2.88	53.50
2.0–4.0	97.06	12.20	96.52	0.86	23.28
Total	99.60	100	50.68	100	100

Table 4
Recovery and grade of copper in the heavy fractions separated from <4 mm vacuum pyrolysis residue particles by gravity separation using a vertical zigzag airflow separator.

Particle size (mm)	Grade of copper (%)	Recovery of copper (%)
<0.105	37.23	53.26
0.105–0.28	60.37	82.75
0.28–0.45	95.66	95.63
0.45–0.9	99.31	100
0.9–2.0	99.64	100
2.0–4.0	99.58	100
Total	99.50	99.86

size due to its ductility or malleability. And the distribution percentage of copper reduced as the particle size decreased. On the contrary, the distribution percentage of non-metallic components increased with decreasing particle size of the crushed vacuum pyrolysis residues of WPCB. The distributions of copper clearly differed from those of the non-metallic components, indicating different crushing characteristics in the high-speed shearing machine. This happened because there was a higher difficulty in crushing copper than glass fiber and pyrolytic carbon. The experimental results might be related to the distinctive mechanical properties of the constituent parts of the vacuum pyrolysis residues of WPCB in the high-speed universal crusher.

3.3. Gravity separation

Air classification is an important unit operation for the separation of dispersed solid particles based on their difference in size and density. And air classification is one among the clean mechanical separation methods that can achieve reasonably good separation of metals and plastics from the PCB stuff [9]. Moreover, air classification is a cleaner separation method that does not use any polluting medium for separation. The principle of pneumatic separation is based on the fact that the particles suspended in a flowing gas, usually air move towards different points under the influence of different forces so that they can be separated from one another. Particles experience gravity and drag forces acting in opposite directions. Heavy particles, having terminal settling velocity larger than the velocity of air move downwards against the air stream, while the light particles whose terminal settling velocity is smaller than the velocity of air rise along with the air stream to the top of the column.

Gravity separation was carried out using a vertical zigzag airflow separator to separate the copper and non-metallic components of the crushed vacuum pyrolysis residue of different particle sizes: <0.105, 0.106–0.28, 0.28–0.45, 0.45–0.9, 0.9–2.0 and 2.0–4.0 mm. The air velocities in the zigzag channel were pre-determined as: 1.1 m/s for <0.105 mm, 1.3 m/s for 0.105–0.28 mm, 1.5 m/s for 0.28–0.45 mm, 1.8 m/s for 0.45–0.9 mm, 2.5 m/s for 0.9–2.0 mm, and 3.3 m/s for 2.0–4.0 mm, at a constant feed rate of 150 g/min. The results of pneumatic separation of the copper from the crushed vacuum pyrolysis residues were shown in Table 4. The separation of the copper into the heavy fractions increased with increasing particle size of the crushed vacuum pyrolysis residue. Almost all the copper were separated into the heavy fractions from the crushed vacuum pyrolysis residue of size >0.45 mm, and 82–95% of the copper were separated into the heavy fractions from the crushed vacuum pyrolysis residue of size 0.105–0.45 mm, but this was remarkably reduced to 53% as the particle size of the crushed vacuum pyrolysis residue became smaller than 0.105 mm. Since the amounts of copper in the particles of size <0.45 mm, especially in the particles of size <0.105 mm, based on the whole WPCB were very small, it was inconsequential that the efficiencies of the separation were lower for copper in these particle sizes.

It was observed from Table 4 that the grade of the copper was improved slightly from 84–97% to over 99% by the pneumatic separation of >0.45 mm crushed vacuum pyrolysis residue. This suggested that inconsiderable amounts of non-metallic components were still introduced into the heavy fraction, which might be attributed to sufficient liberation of the copper and non-metallic components in the crushing of the vacuum pyrolysis residue using the high-speed shearing machine. However, for the crushed vacuum pyrolysis residue of size <0.45 mm, the grade of the copper was improved significantly from 0.04–5.05% to 37–96% by the gravity separation, but this result was insignificant due to the inconsiderable amounts of copper in the particles of size <0.45 mm based on the whole PCB (see Table 3). The grade of copper was 99.50% in the heavy fractions and the cumulative recovery of copper was 99.86% based on the whole WPCB (see Table 4). Therefore, the separation of the copper could be achieved efficiently by the gravity separation using a vertical zigzag airflow separator. Moreover, the glass fiber could be obtained from the non-metallic components after calcinations in a muffle furnace at 600 °C for 10 min (see Fig. 4).

4. Conclusions

A new recycling process without negative impact to the environment for recycling WPCB was established. It was found that the recycling process technology containing vacuum pyrolysis, crushing, size classification and gravity separation was an efficient method to recycle the different fractions of WPCB such as organic resins, copper and glass fiber. The following conclusions could be drawn from the present study.

Vacuum pyrolysis of the WPCB led to an average mass balance of 74.7 wt.% residues, 15.0 wt.% oils, and 10.3 wt.% gases. The residues mainly consisted of copper, glass fiber and carbon. And the residues were very friable and better laminated that could be easily liberated for copper recovery. The oils were a mixture of organic compounds, which were mainly composed of phenol, substituted phenols, benzofuran and substituted benzofurans as well as bromophenols. The gases consisted mainly of carbon dioxide, carbon monoxide, C_1–C_4 alkanes and alkenes, as well as methyl bromide and hydrogen bromide. The oils and gases could be used as fuels or chemical material resources after the proper treatment.

A high-speed universal crusher equipped with cutters using cutting stresses was effective for liberating the copper present in vacuum pyrolysis residues of WPCB. The copper liberation degree of 99.60% was obtained in the fractions of size <4 mm. The amount of copper in the fractions of size >0.45 mm was about 99% based on the whole WPCB, while approximately 92% of the non-metallic components were concentrated in the finer fractions with particle size <0.45 mm.

The separation of the copper and non-metallic components from the crushed residues of size <4 mm was achieved efficiently by the gravity separation using a vertical zigzag airflow separator. The copper grade of 99.50% with recovery of 99.86% based on the whole WPCB was obtained. The glass fiber could be obtained from the non-metallic components by calcinations at 600 °C for 10 min.

Acknowledgments

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