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# Printed circuit board waste as a source for high purity porous silica

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ABSTRACT

The electronic waste stream is one of the fastest growing streams of waste in the world. Printed circuit boards which are found in all electronic equipment are composed of a mixture of various metallic and non-metallic constituents. In this manuscript, the nonmetallics have been treated with acids followed by thermal treatment aiming at the production of high purity porous silica. The results show the possibility of obtaining $SiO_2$ with a specific surface area (BET) as high as 300 $m^2/g$ and purity higher than 99% (X-ray fluorescence, molar%). The as-prepared silica is comparable to industrially sold samples. The study has successfully demonstrated the possibility of using these unwanted electronic wastes for the synthesis of materials with higher value and utility.

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## 1. Introduction

With advancements in the electronic world almost occurring on a day-to-day basis and increased availability of products to the public, it is not surprising to see a staggering increase of electronic wastes over the past several decades. According to a United Nations Environment Program (UNEP) alert bulletin in 2005, 20–50 million tons of e-wastes are produced every year with an estimated annual increase rate of 4% [1]. Miniaturization of electronic devices such as mobile phones due to technological advancement does not slow down the growth of the waste stream, as the effect has been counter-balanced by increased sales, particularly in developing countries. The production of electrical and electronic devices is expected to be the fastest-growing sector in industrialized countries even with implemented environmental regulations [2]. The rapid growth of the e-waste stream is due to economic growth, shorter lifespans of electronic devices, and the development of diversified electronic products in recent years [3].

In general, e-wastes consist of more than a thousand different substances [4]. These substances can be categorized into the general categories of metals and non-metals. Manufacturing mobile phones and personal computers consumes 3% of the gold, 2.5% of the silver, and 12% of the palladium mined annually world-wide. 15% of cobalt mined annually across the globe is also incorporated into these devices mainly as a component of the Li-ion battery [5].

In Hong Kong, the amount of Waste Electric and Electronic Equipment (WEEE) generated has been increasing at a steady rate of approximately 2% per year. The amount of WEEE production per person is about 10 kg per year. With a population of over 7 million people, this amounts to more than 70 million kg of e-waste per year. Citizens of Hong Kong are heavy e-waste-producers, even when compared to Americans who produce about 7 kg of e-waste per person per year [6]. For India, South Africa, and Brazil this number is approximately 0.4, 1.3, and 2 kg per person per year, respectively [3,7].

A Printed Circuit Board (PCB) is the generalized term used for the platform upon which microelectronic components such as semiconductor chips and capacitors are mounted. PCBs are used to support the electronic components as well as to connect them using conductive pathways, tracks, or signal traces etched from copper sheets laminated onto them. A PCB can also be referred to as a printed wiring board (PWB) or etched wiring board. A PCB populated with electronic components is sometimes referred to as a printed circuit assembly (PCA), also known as a printed circuit board assembly (PCBA).

Before PCBs can be processed, they must be disassembled and separated from the body of the waste electronic equipment. Based on the fact that WEEE comes in various different shapes and sizes, it is difficult to develop a mechanized process for disassembling PCBs from all WEEE. Thus, at present, manual disassembly is the

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most practical option. There are several research projects underway for developing automated and semi-automated processes for removing the different components from PCBs [8–10].

It should be noted that the components of circuit board assemblies are held on to the board via solder; so effective recovery of solder via electrochemical or thermal methods would in turn lead to the separation of the components from the board itself [11–15]. An alternative could be to use molten salts to dissolve glasses and oxides, and to destroy plastics present in wastes without oxidizing the most valuable metals [16]. The removal of valuable metals such as palladium [17], gold [18], and silver [19] from WPCBs have been studied. Due to its large content, the separation of copper has always been a popular target for researchers [20–24].

In a review in 2009, Guo et al. [25] reported that although the major economic driving force for recycling PCBs is the metallic fraction, the non-metallic fraction makes up approximately 70% weight of the PCBs. Thermosetting resin matrix composites, thermoplastic matrix composites, and concrete and viscoelastic materials [26] were identified as the main physical recycling routes of the non-metallic fraction. Supercritical fluids depolymerization, hydrogenolytic degradation, as well as other thermo-chemical processes have also been tested [27–31]. In one creative study, acrylonitrile–butadiene–styrene waste plastic has been co-recycled with the nonmetal particles from WPCBs to manufacture composites [32]. The recovery of oils and other compounds from printed circuit board pyrolysis has recently been reported [33–35]. Low-value applications such as being used as fillers have also been investigated [36,37]. Our research group has previously used the non-metallic fraction of WPCB for the production of a novel adsorbent for wastewater treatment [38–40]. In the current paper, for the first time, a simple process for the production of amorphous porous silica from the non-metallic fraction of WPCB is reported. The porous silica can in turn be used in countless applications [41–43].

## 2. Materials and methods

The non-metallic fraction of printed circuit boards was collected from Total Union PCB Recycle Ltd. The waste printed circuit board (WPCB) material consists of a powdered mixture of cured thermosetting resins, glass fiber (cellulose paper), ceramics, brominated flame retardants, residual metals and other additives [25]. This paper is concerned with the removal of the residual metals and volatile fraction of the non-metallic WPCB.

Hydrochloric acid (37%), nitric acid (70%), and sulfuric acid (95%) were purchased from Sigma Aldrich and diluted to the desired molar concentration. The e-waste was treated with various acids for different durations of time and temperatures in a round-bottom flask. Typically 4.0 g of WPCB was used in a 100 mL solution. The treated sample was then filtered, washed, and dried. In order to remove the bromine and volatile fraction, subsequent heat-treatment was carried out in a muffle furnace.

The Materials Characterization and Preparation Facilities (MCPF) at the Hong Kong University of Science and Technology assisted in the X-ray Fluorescence Spectroscopy (XRF) and X-ray diffraction (XRD) analyses. A JEOL JSX-3201Z model automatic sequential XRF was used. The XRD patterns were obtained via a Philips powder diffraction system (model PW 1830) using a Cu Kα source operating at 40 keV at a scan rate of 0.025 $s^{-1}$.

Nitrogen adsorption–desorption isotherms were measured using a Quantachrome Autosorb-I Surface Area Analyzer. The dried samples were outgassed at 150 °C followed by nitrogen sorption at −251 °C. The Brunauer–Emmett–Teller (BET) and t-plot equations were used for calculating the total surface area and the micropore volume respectively. The total pore volume was calculated as the volume of nitrogen adsorbed at $P/P_0 = 0.9814$. The mesopore volume could subsequently be calculated as the difference between the total and micropore volumes. The BJH method was used for discerning the pore size distribution.

## 3. Results and discussion

In order to obtain an initial understanding of the non-metallic fraction of the WPCB, characterization was carried out. The elemental carbon, hydrogen, nitrogen, and sulfur (CHNS) and X-ray florescence (XRF) analyses data are shown in Tables 1 and 2, respectively. Note that the XRF data is relative (sums to 100% even though it does not include lighter elements such as carbon and oxygen), while the CHNS is an absolute mass fraction of the WPCB.

The relatively high bromine content is due to the existence of brominated flame retardants. Materials with low flammability are required for PCBs due to the risk of rising temperatures during assembly and operation of the board. Although phosphorous or nitrogen based fire retardants are available as an alternative and post-industrialized countries are gradually moving towards ending halogenated fire retardants in PCBs, brominated fire retardants are still overwhelmingly predominant on a global scale [45]. Some of the most common types of brominated flame retardants include polybrominated biphenyl, polybrominated diphenyl ethers, and hexabromocyclododecane. Fig. 1 shows the most common brominated flame retardant used in printed circuit boards, namely tetrabromobisphenol A (also known as 2,2′,6,6′-tetrabromo-4,4′-isopropylidenediphenol or more simply TBBPA) [46].

### 3.1. Nitric acid treatment

From among commonly encountered acids, silica is only soluble in HF. Hence, it should be possible to remove the remnant metals in the non-metallic fraction of the WPCB through reactions with acids which do not dissolve silica. Since metal nitrates are known to be ubiquitously soluble, the first acid to be tested was nitric acid. Table 3 shows the effect of temperature on the metal removal. As temperatures rise from 50 to 70 °C a significant decrease in calcium concentration is seen. Further increase of the temperature, enhances the calcium removal but to a lesser extent.

Since the slurry boils at 110 °C, the temperature cannot be increased further under atmospheric pressure. Table 3 shows that

**Table 1**
CHNS elemental analysis of the non-metallic fraction of WPCB [44].

| Element | C | H | N | S | Others (by difference) |
|---|---|---|---|---|---|
| Weight% | 21 | 0.1 | 0.6 | 0 | 78.3 |

**Table 2**
XRF elemental analysis (relative) of the non-metallic fraction of WPCB.

| Element | Al | Si | S | Ca | Ti | Fe | Cu | Br | Ba |
|---|---|---|---|---|---|---|---|---|---|
| Molar% | 8 | 51 | 1 | 30 | <1 | <1 | 3 | 5 | <1 |

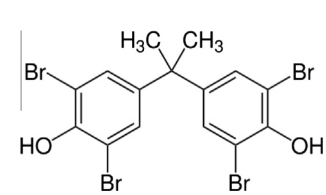


**Fig. 1.** Structure of tetrabromobisphenol A.

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