

CHAPTER 3

Pretreatment of WPCBs

3. Pre-treatment of WPCBs

3.1 Hammer milling of WPCBs

The shortened WPCBs has been milled to -16 BSS using hammer mill under varying feed size, feed rates and time. The results have been discussed in details.

3.1.1 Effect of feed size of WPCBs on milling

To study the effect of feed size on the milling of WPCBs to -16 BSS, the preliminary experiments were carried out at the feed rate $3 \text{ kg}\cdot\text{h}^{-1}$ for 7 minute. The shortened WPCBs of different sizes of $1 \times 1 \text{ cm}^2$, $1.5 \times 1.5 \text{ cm}^2$, $2 \times 2 \text{ cm}^2$, $2.5 \times 2.5 \text{ cm}^2$ were fed into hammer mill to find out the optimized feed size of WPCBs. The effect of feed size on milling of WPCBs (%) has been shown in figure-3.1. WPCBs size more than $2.5 \times 2.5 \text{ cm}^2$ were not found appropriate for feeding through the maximum size of inlet chute of hammer mill. Therefore, the initial experiments were started with $2.5 \times 2.5 \text{ cm}^2$. The larger sizes basically took more time to induce brittle fracture of glass and ceramics part. Hence, it was found that 69.50% of WPCBs were milled with feed size $2.5 \times 2.5 \text{ cm}^2$. Further, the size reduction from $2.5 \times 2.5 \text{ cm}^2$ increases the milling rate and 84.25%, 94.35% WPCBs were milled at feed size $2 \times 2 \text{ cm}^2$, $1.5 \times 1.5 \text{ cm}^2$ respectively. The maximum milling (99.30%) of WPCBs were achieved with feed size $1 \times 1 \text{ cm}^2$. Hence, $1 \times 1 \text{ cm}^2$ was chosen as optimized feed rate for further studies.

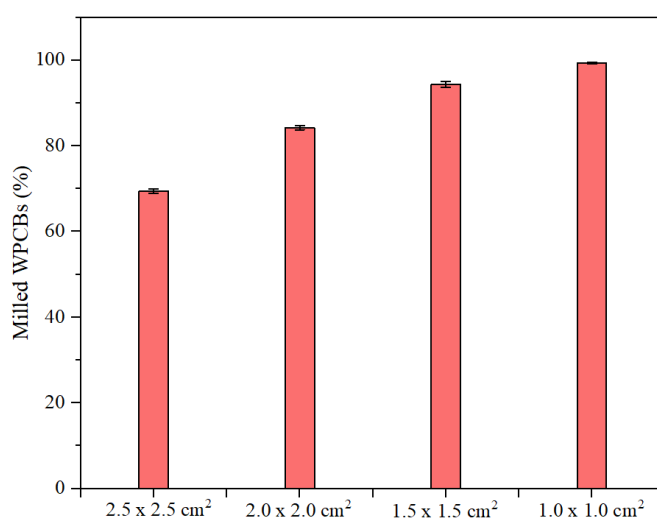


Figure 3.1- Effect of feed size on milling of WPCBs

3.1.2 Effect of feed rate and time on milling of WPCBs

WPCBs of optimized feed size; $1 \times 1 \text{ cm}^2$ were fed into the hammer mill with various feed rate such as 2,3,4,5 kg.h^{-1} to find out the optimized feed rate. The combined effect of milling time and feed rate on milling of WPCBs has been shown in figure-3.2. Increased feed rate of WPCBs from 2 to 5 kg.h^{-1} led to 29 % reduction in the milling rates of WPCBs crushed to -16 BSS sieve size. The one of the reason behind decreased milling rate at higher feed rates is the reduction in hammer's impact velocity due to the resistance to movement of hammers. Moreover, the higher feed rates also result in the distribution of applied impact energy among more pieces of WPCBs. The forces under this circumstances were not sufficient to fracture the non-metallic part of WPCBs and the impact energy is absorbed by the pieces of WPCBs. Eventually, this deteriorates the milling rate and also cause overheating of WPCBs. In most of the cases, the milled fractions were found constant after 7 min of milling time and maximum milling efficiency was seen with feed rate 3 kg.h^{-1} . WPCBs with feed size $1 \times 1 \text{ cm}^2$ and feed rate 5 kg.h^{-1} were 66.36% milled within 8 min. However, 99.30% of WPCBs were milled within 7 minutes with decreased feed rate of 3 kg.h^{-1} . Therefore, the optimized feed rate and milling time was chosen 3 kg.h^{-1} and 7 minutes for further studies.

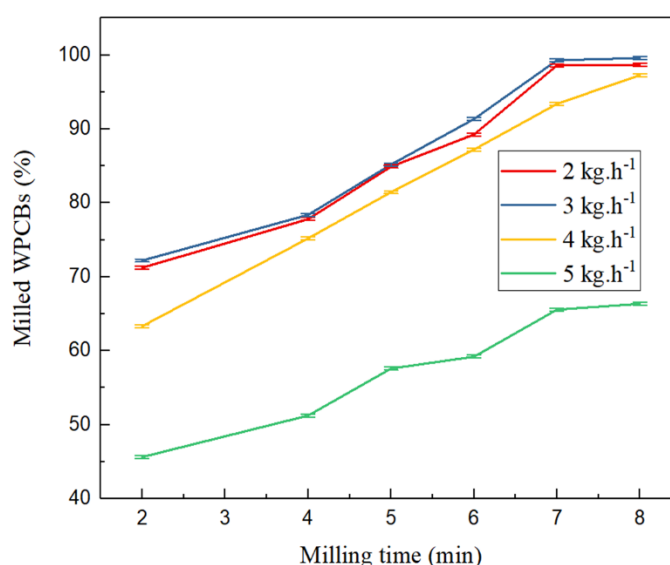


Figure 3.2 - Effect of feed rate and milling time on milling of WPCBs

3.2 Ultra sonication treatment of hammer milled WPCBs

The hammer milled WPCBs of size -16 BSS were sent for further concentration of metallic fractions using ultra sonication treatment. Due to the cavitation effect under the ultrasonic treatment, the generation of micro jets and shock waves with in the pulp of WPCBs and water also get increased and drives the particles in the liquid to move uninterruptedly and strike with each other, which promotes the detachment of metallic substances from the WPCBs during the ultra-sonication as shown in the figure-3.3. The effect of pulp density and ultra-sonication time has been studied to find the optimized ultra-sonication parameters.

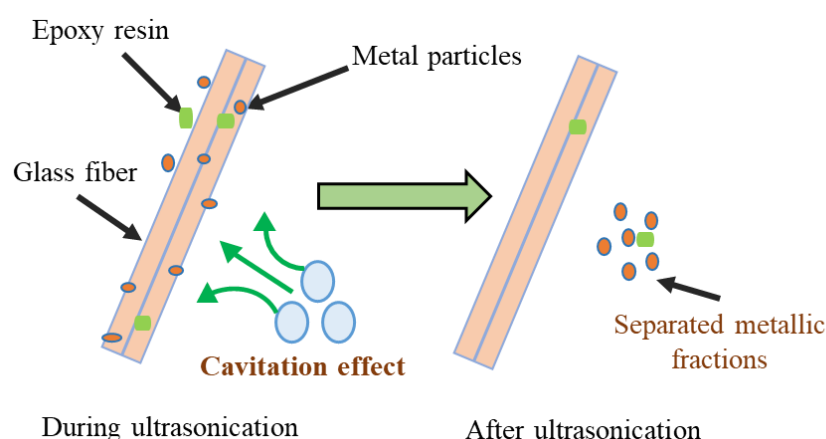


Figure 3.3- Mechanism of ultrasonic-milled WPCBs interaction

3.2.1 Effect of pulp density on separation of metallic fractions

The solid to liquid ratio plays an important role during the ultra-sonication of milled WPCBs. Therefore, the effect of pulp density on separation of metallic fractions has been studied and shown in figure-3.4. The separation efficiency was found lower at low value of pulp density such as 2×10^{-2} , 5×10^{-2} g/mL and maximum (41.35%) at 10×10^{-2} g/mL. Further, increase in pulp density decreases the % separated metallic fractions. The increase in pulp density basically forms lumps and that decreases the wettability of WPCBs particle. Moreover, the liquid jets and micro shocks generated at high pulp density was not sufficient to disperse the metallic

fractions from the WPCBs and water mixture. Therefore, 10×10^{-2} g/mL was chosen as optimized pulp density for the maximum separation of metallic fractions from the milled WPCBs.

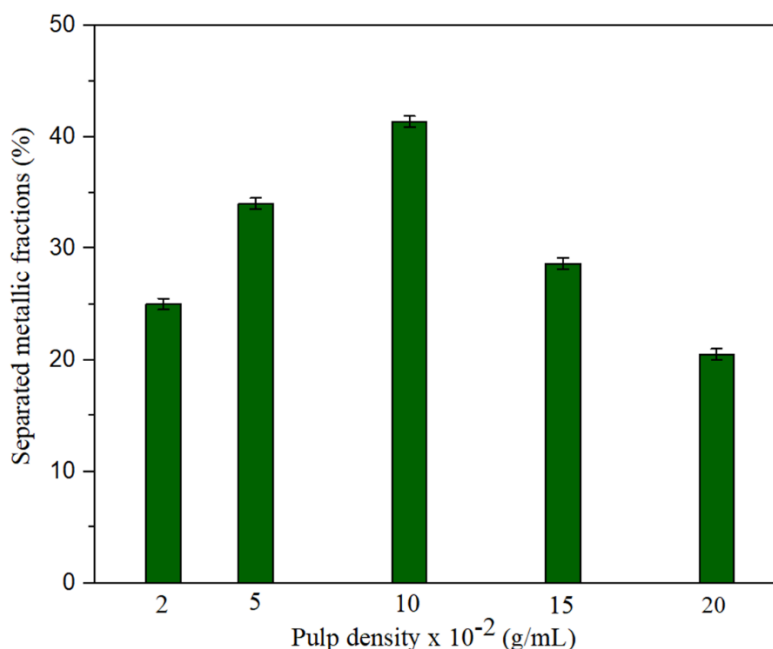


Figure 3.4- Effect of pulp density on separation of metallic fractions

3.2.2 Effect of ultra-sonication time on separation of metallic fractions

The effect of time has been studied with 10g WPCBs milled sample at 10×10^{-2} g/mL at varying time for 2,5, 10, 15, 20,25 and 30 min and is shown in figure-3.5. In the case of 2,5,10 min of ultra-sonication time, the separated metallic fractions were around 0.6, 1.03, 2.315 grams respectively. However, in the case of 15 and 20 min, the metallic fractions obtained were somewhat at satisfactory level, around 3.458 and 4.135 g. Moreover, further increase in ultra-sonication time (25,30 min) does not show any effect on the separation of metallic fractions and it was concluded that 20 min ultra-sonication time is sufficient to separate metallic fractions from the milled WPCBs. The complete separation of metallic fractions was seen at 20 min ultra-sonication time with 10×10^{-2} g/mL pulp density.

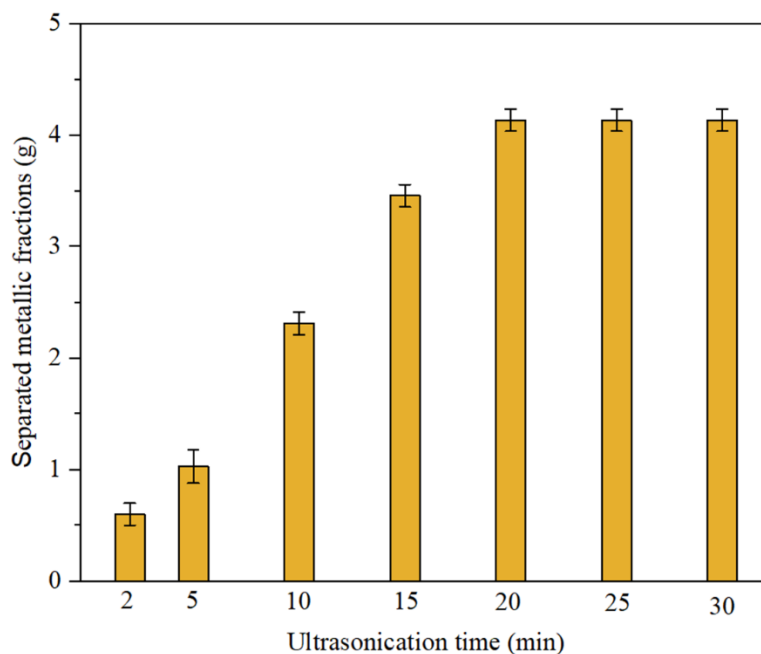


Figure 3.5- Effect of ultra-sonication time on separation of metallic fractions

3.3 SEM-EDS and XRD analysis

WPCBs powder obtained after hammer milling and the metallic fractions separated by ultrasonic treatment are characterized by SEM-EDS to check these powders' particle morphology and elemental distribution. The sample of raw WPCBs (hammer milled), its SEM micrograph, and its chemical composition has been shown in Figure-3.6a. The SEM micrograph of raw WPCBs powder shows a mixture of irregular flaky along with the needle shape fibrous structure. This confirms the existence of epoxy resins and glass fiber within the raw WPCBs powder. The EDS spectrum shows the presence of various metals (base, precious, toxic) along with the non-metallic fractions.

The sample of metallic fractions separated by ultrasonic treatment and its SEM-EDS has been shown in Figure-3.6b. The SEM micrograph shows the particles of metallic copper, and no needle shape or flaky structure has been observed. This confirms the separation of non-metallic fractions from the raw WPCBs powder after employing the ultrasonic treatment. Moreover, the

EDS spectra show the copper in maximum percentage. Other base and precious metals have also been observed in the EDS of metallic fractions.

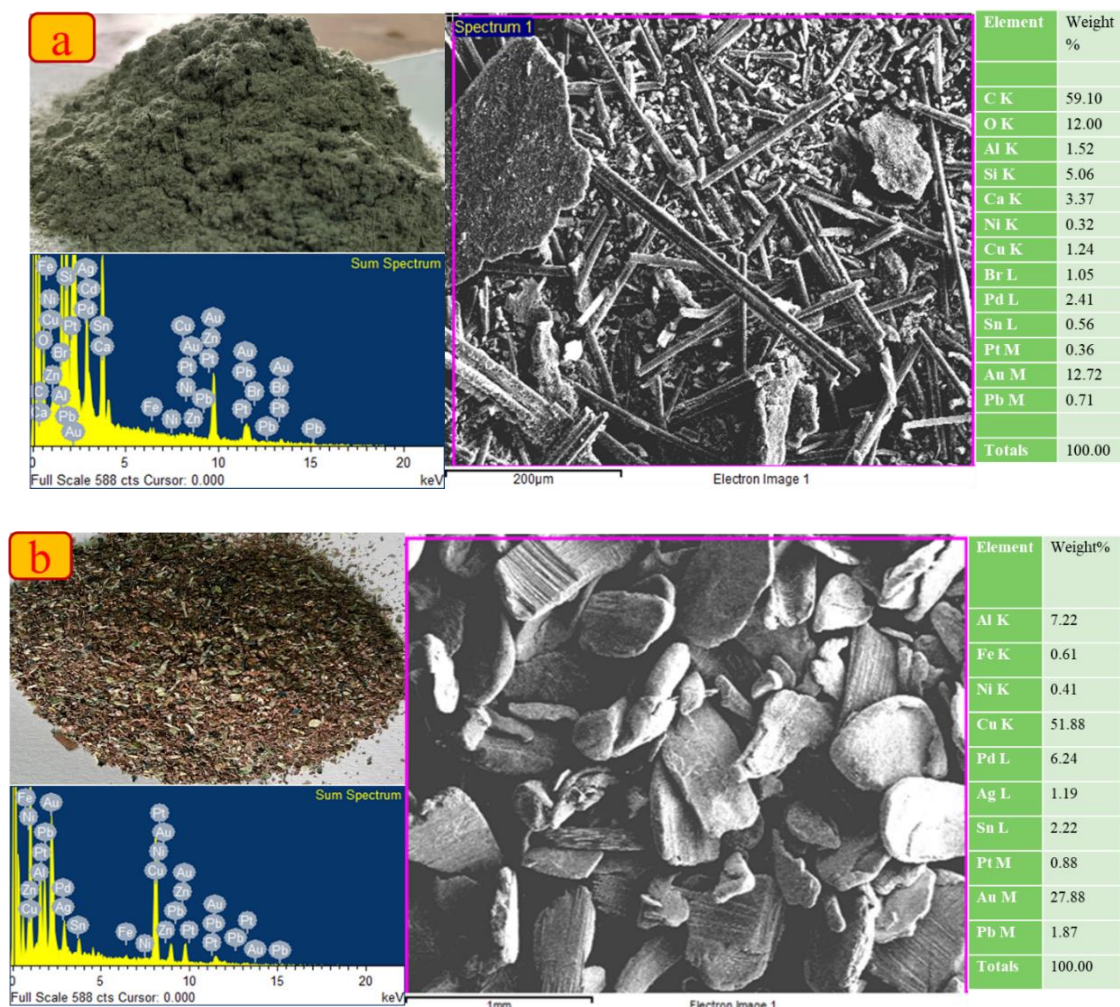


Figure 3.6- SEM micrograph and EDS analysis of (a) Raw WPCBs powder, (b) ultrasonic treated WPCBs metallic fractions

The XRD pattern of raw WPCBs powder and ultrasonic pre-treated metallic fractions has been shown in Figure-3.7 (a, b). The amorphous phase and low peak intensities were recorded in feed in the range of 20-35° along with relatively higher intensity peaks of copper (50.725°, 59.288°, 88.770°). However, the amorphous and low-intensity peaks are not visible in ultrasonically cleaned metallic fractions. However, metallic copper peaks with higher peak intensities are visible at 50.725°, 59.288°, and 88.770°. This also indicates the parting of metallic and non-metallic portions.

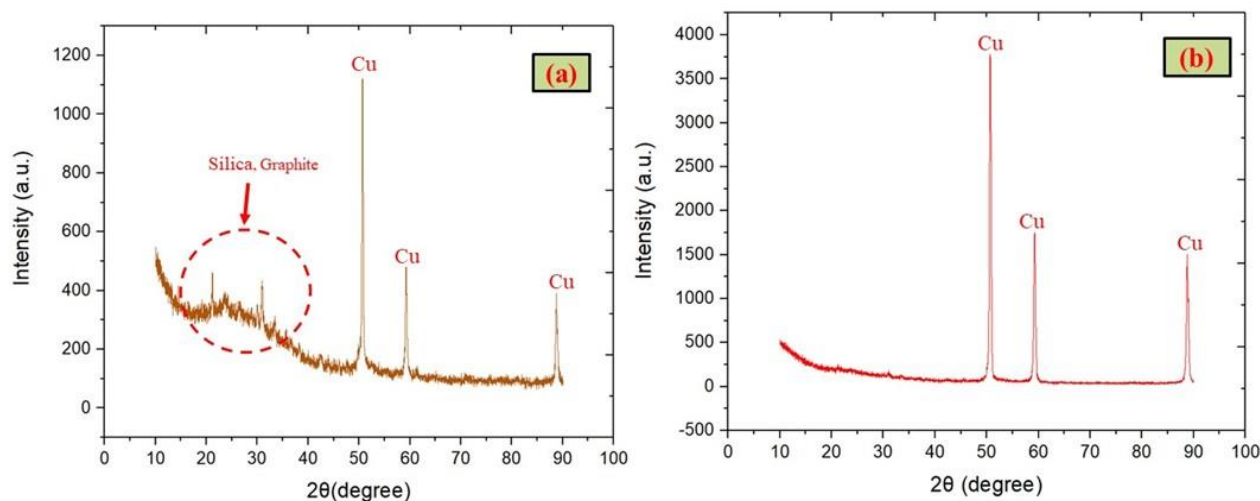


Figure 3.7- XRD patterns of (a) Raw WPCBs powder and (b) ultrasonic treated metallic fractions

3.4 FTIR analysis of Raw WPCBs and ultrasonically separated non-metallic fractions

Fourier Transform Infrared Spectroscopy (FTIR) basically provides us with information regarding the functional groups of compounds. In our work, we recorded infrared spectra of raw WPCBs powder and ultrasonically cleaned WPCBs powder as shown in Figure-3.8. The band at 3462 cm^{-1} indicates the C=O overtone and the band at 2970 cm^{-1} represents the alkanes (Chen et al., 2022). The C=O (carbonyl) functional group was observed at 1740 cm^{-1} , confirming polycarbonate's presence in the WPCBs powder (Haghighi-Yazdi and Lee-Sullivan, 2015). Similarly, the 1615 and 1001 cm^{-1} band represents C=C stretch and tetra-substituted bonds, respectively (Jadhao et al., 2020). The band at 1505 represents aromatic rings.

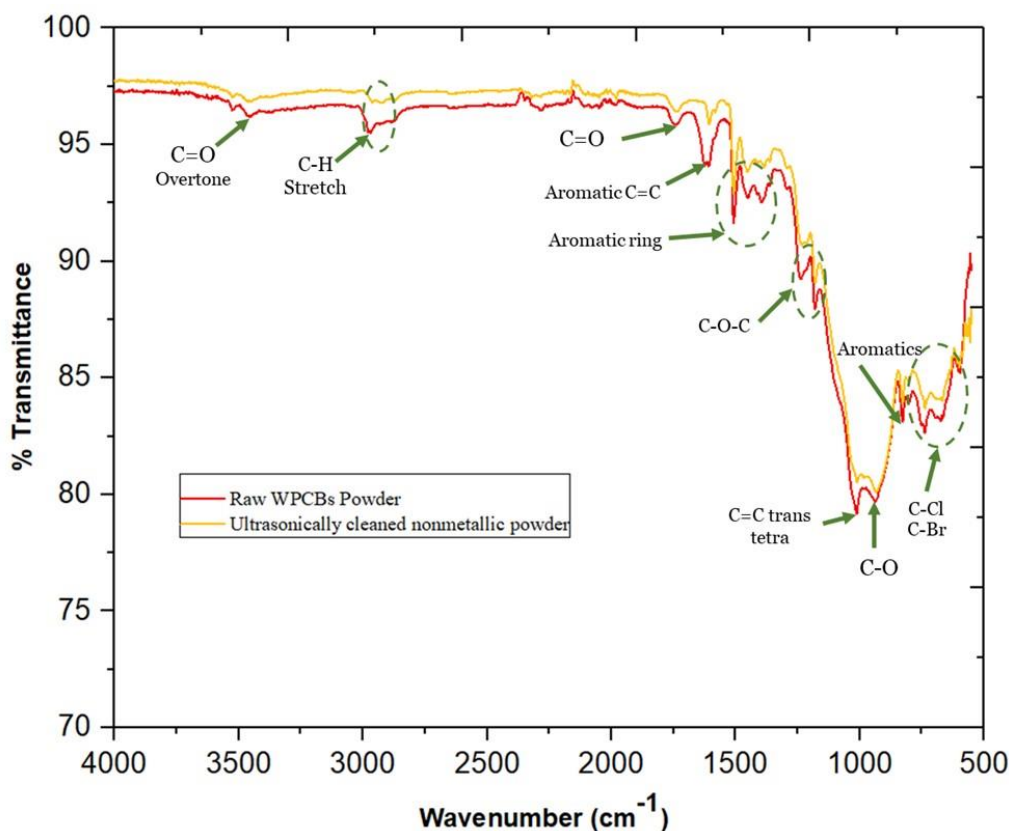


Figure 3.8- FTIR Spectra of WPCBs powder

The C-O-C stretching vibrations were assigned to the band 1179 and 1231 cm^{-1} (Barnwal et al., 2020; Liu et al., 2021). The band at 932 cm^{-1} represents the C-O bond. The band at 729 cm^{-1} is assigned as C-Cl, confirming the presence of polychlorinated biphenyls, and those at 660 cm^{-1} represent the C-Br (Reena et al., 2011). The band at 660 cm^{-1} confirms the existence of flame retardants (polybrominated biphenyls) used in WPCBs (W. Chen et al., 2021). The FTIR confirms that the WPCBs powder contains various polymers, glass fiber, and brominated epoxy resins. The IR spectra of ultrasonically separated non-metallic fractions of WPCBs are observed to check the changes occurring during the ultrasonic treatment. However, no new or extra peaks are observed on the IR spectra. This confirms that no chemical reaction took place during the ultrasonic treatment of WPCBs powder and no sign of any other bonding. Hence, ultrasonic treatment may only be characterized under the combination of mechanical and physical beneficiation processes.

3.5 Chemical analysis

Due to the creation of high-speed liquid jets, the lighter particles go upward while the heavy metal particles settle and agglomerate during ultra-sonication. The effective separation of metallic powder was seen after 20 min of ultra-sonication. The metal content of the raw WPCBs powder and separated metallic powder has been shown in table-3.1. From this, it can be concluded that the ultrasonic irradiation treatment is an efficient process to concentrate the targeted metal and for separating the non-metallic part from the mixture of metallic and non-metallic fractions. Taking an example of copper, it is 51 wt% copper when compared to directly milled powder.

Table 3.1-Chemical analysis of Raw WPCBs powder, ultrasonically cleaned metallic powder

Element	Weight % of elements	
	Raw WPCBs powder	Ultrasonic-treated Metallic fractions
Copper	42.73	87.00
Nickel	0.40	2.67
Gold	0.0013	0.032
Silver	0.08	0.063
Palladium	0.003	0.007
Tin	1.33	0.600
Iron	0.32	0.430
Zinc	0.23	0.400
Lead	0.08	0.067
Cadmium	0.11	0.220
Plastic and Ceramic Residue	54.60	8.51

Conclusions

- 99.3% of WPCBs were milled within 7min at optimized feed rate ($3\text{kg}\cdot\text{h}^{-1}$).
- Successfully demonstrated ultra-sonication of hammer milled WPCBs to separate metallic fractions.
- The effective separation of metallic fractions was seen after 20 min of ultra-sonication with 10×10^{-2} g/mL pulp density.
- The higher concentration of elements such as copper and nickel were observed in the ultrasonically cleaned metallic fractions.
- The first ever studies on direct ultra-sonication of hammer milled WPCBs without using any toxic solvent and thermal treatment.