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A novel approach to epoxy coating removal from Waste Printed Circuit Boards by solvent stripping using NaOH under autoclaving condition

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ABSTRACT

The Printed Circuit Board (PCB) was an insulating material as a whole, but having conductive lines printed over it or etched from it and found virtually in all sorts of Electrical and Electronic Equipment (EEE). To process the PCB in an environmentally sound manner, various techniques were under research to reduce the toxicity in the treatment process. Toxicity can be reduced by removing the epoxy coating from the PCB. In our present investigation, solvent stripping combined with autoclave treatment at 121 °C temperature under a pressure of 1.1 kg/cm^2 results in 100% epoxy removal efficiency for concentration varying from 0.25 N to 4 N of Sodium hydroxide. Epoxy was completely peeled off from WPCB at a treatment time of 1 h at 0.25 N of Sodium hydroxide prior to recycling WPCB for its metals. Sodium hydroxide was regenerated by filtering the solution through Whatman No.1 filter paper of 125 mm diameter circles having a pore size of 11 μm using vacuum filtration. Sodium hydroxide regeneration was analyzed with the help of Fourier Transform Infra-Red spectroscopy analysis and the metals like copper and lead were leached from WPCB during the pre-treatment process in the NaOH solution was analyzed using Atomic Absorption Spectroscopy.

1. Introduction

Electronic waste or e-waste refers to all sorts of electrical equipment as well as electronic equipment and gadgets reached its end of its life (Yaazhmozhi et al., 2020). Printed Circuit Board was a non-conducting material found in all sorts of Electrical and Electronic Equipment (EEE), here the electrical interconnections were provided to the PCB with the help of micro-electronic components which includes Integrated Circuits (ICs), Semiconductor chips, Capacitors and Resistors mounted over it (LaDou, 2006). PCB comprises of polymers & metals in which polymers holds more than half of its composition and the rest being metals. Recycling the complex WPCB for metals from it leads to urban mining. For instance, precious metals like gold contribute 200–250 g per tonne of PCB which can be extracted from it by means of urban mining. Urban mining was the recent approach towards the e-waste recycling for its metals because of e-waste has large quantity of metals than next to the metal ores available from natural sources (Arya and Kumar, 2020; Ghosh, 2020; Ravindra and Mor, 2019). Heavy metal accumulation occurred in the environment due to the open disposal of the hazardous e-waste. Earlier treatment ought to be made to the e-waste for eliminating the harmful epoxy covering over PCB and furthermore for heavy metal recycling before it had been discarded into the environment.

Epoxy coating or organic coating applied to the PCB to fortify the sturdiness and also to resist the boards from chemical attack and corrosion. Epoxy being flammable hence bromine was added to coat the PCB from a range of 5% to 15% depending on the purpose of the PCB it provided the equipment to make it as a flame retardant (Senophiyah-Mary et al., 2018). WPCB was a composite structure made up of polymers and metals in which different exploration have been done to eliminate the harmful epoxy covering over the boards. Pyrometallurgy was a process wherein low temperature examination of WPCB prompts the progression of dioxins and furans though the high temperature treatment will oppress the formation of dioxins (Wang et al., 2017). The high temperature examination of WPCB prompts high energy utilization and this system of metal recovery would become uneconomical (Balaji et al., 2019; Balaji and Senophiyah-Mary, 2020; Ma, 2019; Taylor et al., 2015). Some other treatment techniques like hydrometallurgy involving chemical treatment could be unsafe because of the presence of bromine to make the combustible epoxy as fire resistant. Pyrolysis and vacuum Pyrolysis strategies involving metal recovery process lead to outflow

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of dioxins and furans in the environment (Bidini et al., 2014; Long et al., 2010). Crude methods such as incineration in burning in open fields lead to the increase in harmfulness of the air atmosphere (Sohaili et al., 2012). An environmentally friendly approach to the metal recovery from WPCB by bioleaching process was time consuming than usual methods. This was due to the existence of organic epoxy coating between the metallic part and bacteria, which acts as a protective layer and prevents the bacteria from consuming the metallic part of WPCB (Mary and Loganath, 2019; Senophiyah-Mary et al., 2018). Another eco-friendly approach towards the metal recovery was synthesising Nanoparticles from the leach solution of WPCB using the plant leaf extract (Shankar et al., 2004). The similar significant disadvantage during the green synthesis of Nanoparticle was the organic epoxy layer; consequently it had to be completely removed from the WPCB. Numerous researches had been done to eliminate the toxic epoxy layer from the WPCB alongside the need to reduce the toxicity in metal recovery process. Solvents like Dimethylacetamide, Dimethylformamide, methanol and chemicals like sodium hydroxide, hydrogen peroxide and sodium sulphate had been used to break the bonds between the polymeric compound (i.e.,) epoxy coating. Verma et al., (2016) carried out a research in which solvents like Dimethylformamide were used to peel off the Brominated Epoxy Resin (BER) from the surface of the Printed Circuit Boards. When PCBs were treated for the solid–liquid ratio of 300 g/l in Dimethylformamide (DMF) at a temperature of 165 °C for 4 h, the BER was peeled along with the liberation of metal clads from the circuit boards. Later on Verma et al., (2017) carried out a research in removing the organic epoxy layer that was the Halogenated Epoxy Resin Substrate (HERS) adhered to the surface of the Printed Circuit Boards. When PCBs were soaked and treated in Dimethylacetamide (DMA) solution, the HERS were peeled off completely from the surface and dissolved in the solution at a temperature of 160 °C. Regeneration efficiency for the solvents like DMA and DMF was more than 95% after the treatment process. These techniques were feasible and energy proficient for eliminating the HERS, BER and not reasonable for different composition of epoxy resin. Integrated Circuits (ICs) treated under the Super Critical Water condition of temperature 500 °C at a pressure of 23 MPa for a treating time of 90 min; commercially valuable metals such as gold, silver and copper were separated from it (Li et al., 2019; Li and Xu, 2021). In addition to that, epoxy layer adhered to ICs were disintegrated and get dissolved in water. Phenol and derivates of phenol were retained as the liquid products. Conventional method like solvent stripping was the employed to remove the epoxy coating from Waste Printed Circuit Board (WPCB) but it requires chemical solutions of higher concentrations and also it was time consuming. Over few decades Sodium hydroxide was used as stripping agent for removing the epoxy resin. Solder mask adhered to the surface of the WPCBs were stripped by treating the samples soaked under the aqueous NaOH of 5 N for 8 h and later on treated it with bath sonication for 5 min (Senophiyah-Mary et al., 2018). Regeneration of NaOH was confirmed with the help of FTIR spectroscopy when the treated aqueous NaOH solution was filtered using the Whatman No.1 filter paper (Senophiyah-Mary et al., 2018). The objective of this study was to suggest a novel method to remove the organic epoxy coating from WPCB which should be an economically feasible method, as well as to reduce the quantity of spent chemicals or solvents for the removal process. Optimisation studies have been carried out to know the removal efficiency of epoxy coating from WPCB by varying the concentration of sodium hydroxide, soaking time of WPCB after the treatment under autoclaving condition the capacity of autoclave and the quantity of WPCB. To ensure the regeneration of spent sodium hydroxide for the removal of epoxy resin from WPCB using Fourier Transforms Infra-Red spectroscopy and also to quantify the regeneration efficiency. To find the possibility of metals leached during the treatment process was analysed using Atomic Absorption Spectroscopy.

2. Materials and methods

2.1. Collection of WPCB

The WPCB was collected from the college premises Coimbatore Institute of Technology, Coimbatore. WPCB (i.e.,) waste printed circuit board or the waste mother board was taken from the discarded computers and this WPCB was coated on either side by the organic epoxy coating. Components like Integrated Circuits (ICs), capacitors, resistors, semiconductor chips and the slots provided for connecting the output units of the computer were removed from the WPCB; Random Access Memory was also removed from the respective slots of the mother board prior to cutting operations of ICs, resistors, capacitors, other chips and slots (Fig. 1).

2.2. Preliminary process

The WPCB was washed thrice with double distilled water to remove the dirt from the boards. It was then allowed to dry in the heat incubator at 50 °C, after drying the WPCBs were cut into small pieces of size 3 cm × 3 cm for further experimental study.

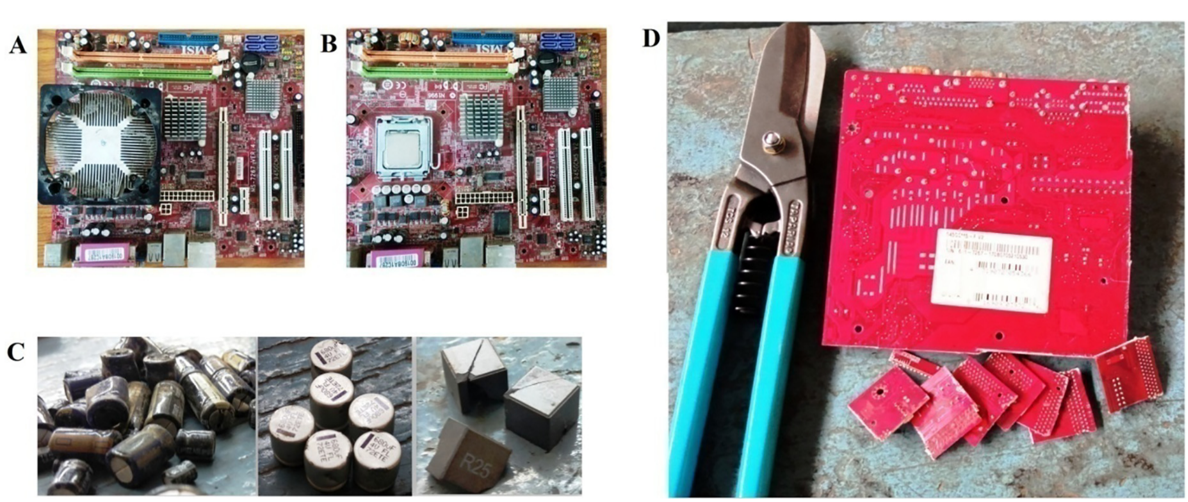

Fig. 1. (A) Collected sample WPCB (B) Dismantling WPCB (C) Dismantled resistors, capacitors from WPCB (D) WPCB plate samples of size 3 cm × 3 cm.

2.3. Sodium hydroxide preparation

Sodium hydroxide was commonly used as a stripping agent to remove the organic epoxy layer from the surface of the PCBs. Sodium hydroxide of different concentrations such as 0.25 N, 0.5 N, 0.75 N, 1 N, 2 N, 3 N and 4 N were used for the experimental study. Salts used for the preparation of sodium hydroxide were of sigma Aldrich grade and the water used for the experiment was double distilled water.

2.4. Experimental procedure

The autoclave was basically an instrument used to sterilize the materials present in the chamber by killing the microbes such as bacteria, virus and spores. To remove epoxy layer from WPCB, the samples were kept in autoclave at different concentrations of sodium hydroxide solution and also in distilled water until the autoclave reaches a temperature of 121 °C and a pressure of 1.1 kg/cm^2. A triplicate set of samples were placed vertically in 100 ml beaker to ensure the surface of the WPCBs completely immersed in chemical solution. To reach the desired pressure of 1.1 kg/cm^2, the time required to attain the pressure in autoclave having a capacity of 55 L was 55 min, 56 min and 45 min for triplicate analysis; this was due to the temperature differences in the water used in autoclave. Once the desired pressure was reached, the steam valve should be opened to wipe out the steam inside the chamber. The samples were taken out and washed in running tap water followed by double distilled water to remove some of the epoxy adhered to the WPCB surface (Fig. 2).

2.5. Fourier Transform Infra-red spectroscopy

The samples were taken out and the solution was filtered using filter paper followed by Whatman No.1 filter paper of 125 mm diameter circles with pore size of 11 μm, then the untreated Sodium hydroxide solution, treated Sodium hydroxide solution and the filtrate were tested in Fourier Transform Infra-red Spectroscopy (Bruker, Model Tensor 27, MA, USA). FTIR was used to identify the functional groups present in the solution and also to figure out the regeneration efficiency of the stripping agent used for epoxy removal (Senophiyah-Mary et al., 2018).

2.6. Atomic Absorption spectroscopy (AAS)

Atomic Absorption Spectroscopy (TL-2800AA/TOPLAB INDIA) was used to find out the metal concentration present in the sample solution. Stock solution of different ppm was prepared for different metal elements like Copper, Lead, Iron, Cadmium and Zinc for the sample analysis. To assess the concentration of leached metals in the NaOH solution before filtration, the samples were tested in AAS to measure it for various components as referenced previously. Samples of two different concentrations such as 0.25 N and 4 N of NaOH solution treated under autoclaving condition were tested in AAS for indentifying the unknown metal concentration. Absorbances for each metal element corresponds to two samples were recorded for quantifying the metal concentration. Calibration curve was drawn for the values of stock solution from which the concentration of metals leached in the treated NaOH solution was identified (Table 1 and 2).

3. Results and discussion

Our proposed method for epoxy removal from WPCB involving solvent stripping under autoclaving condition required 0.25 N of NaOH. In conventional method, 10 N Sodium hydroxide solution was used as a solvent to remove the epoxy from WPCB under static condition. Later on, researchers were trying to reduce the concentration of the NaOH by implementing their innovative ideas. In recent method such as solvent stripping integrated with bath Sonication required 5 N of NaOH and for the conventional method it was 10 N of NaOH. Concentration reduction ratio of NaOH used between the proposed method and the bath Sonication was 1:20 and for the conventional method was 1:40, thus the Concentration reduction was achieved to a great extent. BER was commonly used in recent days to PCBs to ensure the flame retardant property. The reaction mechanism involving the BER and sodium hydroxide treated under autoclaving condition was illustrated below. Forces exerted by the hydrogen of hydroxyl group in parental epoxy resin towards the hydroxyl group of strong base sodium hydroxide contribute the breakdown of hydrogen ions from the epoxy resin. Positively charged sodium ions get attracted towards the negatively charged hydroxyl group of epoxy resin resulted in O-Na bond (Fig. 3).

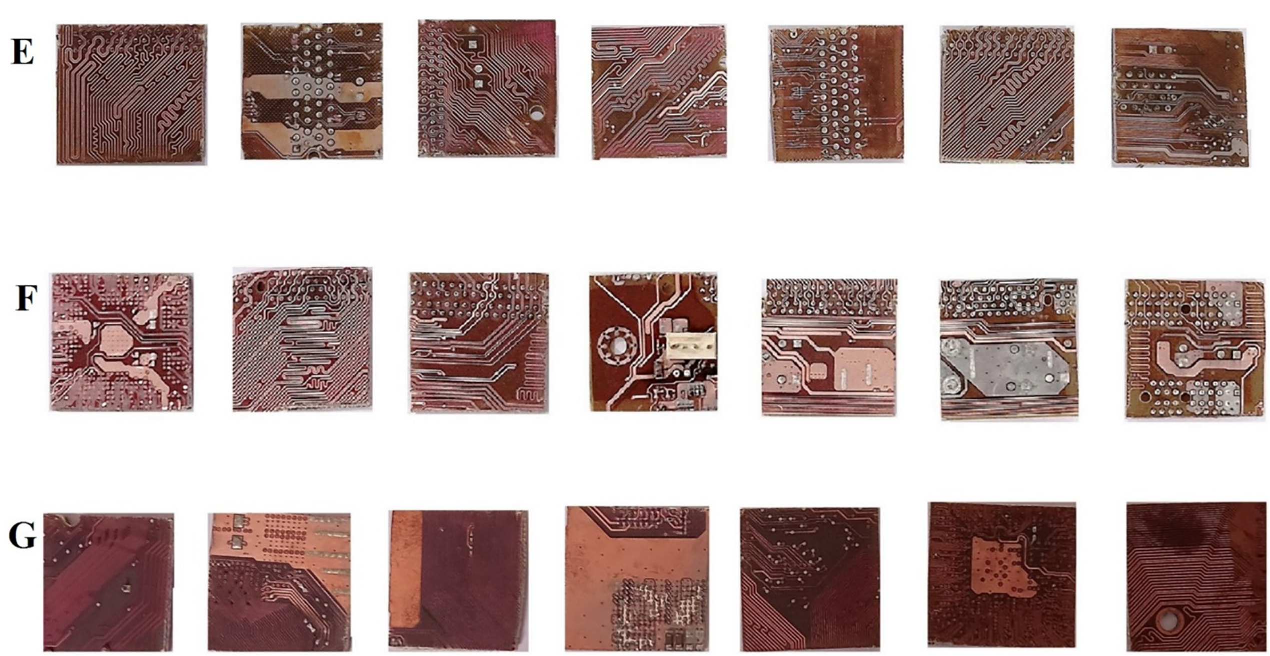

Fig. 2. (E), (F) & (G) Triplicate analysis of WPCB samples treated under different concentrations of NaOH solutions of Normality varying from 0.25 N to 4 N.

Table 1
Optimal instrumental parameters for identifying metals using AAS.

S.No	Metal Element	Sensitive Line (nm)	Operating Current (mA)
1	Copper	324.7	4–10
2	Lead	283.8	5–12
3	Iron	248.3	8–16
4	Cadmium	228.8	5–15
5	Zinc	213.9	5–15

Table 2
Concentration of metal elements for sample before filtration tested using AAS.

S. No.	Tested Metals	Metal Concentration in ppm	
		Sample 1 (0.25 N NaOH treated using Autoclave)	Sample 2 (4 N NaOH treated using Autoclave))
1	Copper	0.416	0.013
2	Lead	0.079	0.079
3	Iron	0	0
4	Cadmium	0	0
5	Zinc	0	0

3.1. Effect of sodium hydroxide concentration

Sodium hydroxide, a strong base was used to remove the epoxy layer adhered to surface of the WPCB. Concentration of Sodium hydroxide played a vital role in breaking the epoxy bond. Increase in concentration of sodium hydroxide for stripping epoxy increased the epoxy removal percentage under a static condition. The main idea behind this experiment was to minimize the concentration of NaOH solution and also to achieve greater removal efficiency in removing the epoxy from WPCBs. WPCB samples were soaked in different concentration of Sodium hydroxide solution varying from 0.25 N to 4 N and kept in autoclave for stripping process. For Sodium hydroxide of higher concentration the epoxy layer gets peeled like a sheet from the WPCB whereas for lower concentration the Sodium hydroxide breaks each particles of the epoxy adhered to the WPCB surface and the disintegrated epoxy was dispersed in the solution thus the NaOH solution acquires the colour of the WPCB. Epoxy gets peeled off completely from the WPCB in autoclave at concentrations of 2 N, 3 N and 4 N in the solution itself and the removal efficiency was 100%. Solutions of lower concentrations such as 0.25 N, 0.5 N, 0.75 N and 1 N also achieved 100% removal efficiency but slight physical energy

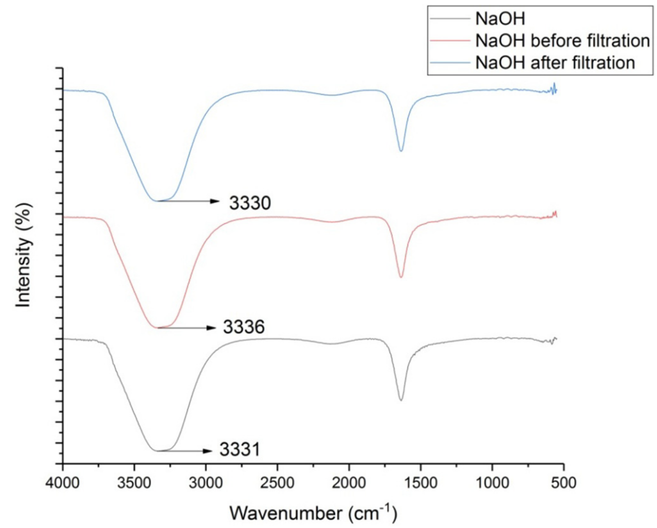

Fig. 4. FTIR spectroscopy analysis for NaOH solution of concentration 0.25 N.

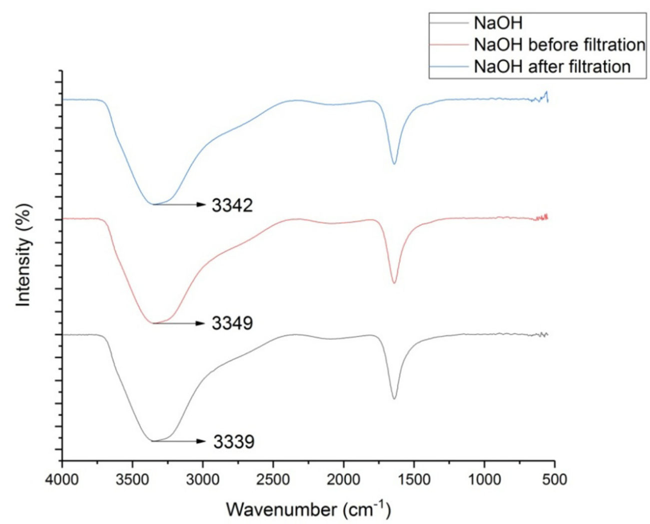

Fig. 5. FTIR spectroscopy analysis for NaOH solution of concentration 4 N.

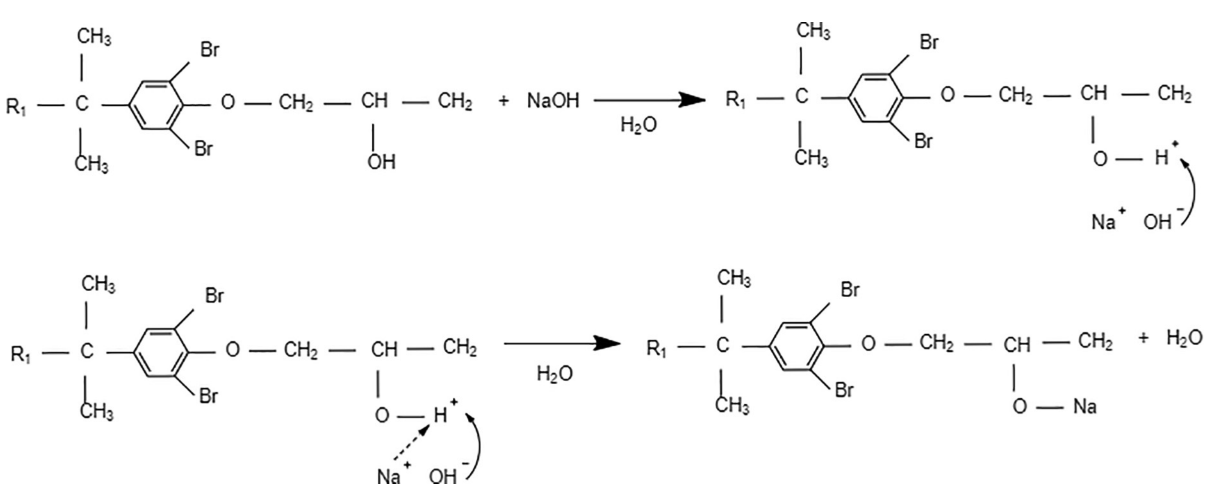

Fig. 3. Hydrogen and Hydroxyl group bond interactions between Brominated Epoxy Resin and Sodium hydroxide.

was needed for scrubbing. For higher concentration such as 2 N, 3 N and 4 N requires a simple tap water wash followed by double distilled water was more than sufficient. Higher concentrated solution doesn't show any colour after the treatment under autoclaving condition this is because the peeled off particles were heavier and it settles down easily whereas for lower concentrated solution the lighter particles dispersed in the solution. A retention time was required for the lower concentrated solution to settle down the dispersed particles. Epoxy removal efficiency was calculated based on the area of epoxy adhered to the surface the WPCB sample by using the following formula.

$$\text{Epoxy removal efficiency} = \left(1 - \frac{\text{Area of epoxy adhered in WPCB sample}}{\text{Area of WPCB sample}}\right) \text{x } 100\%$$

3.2. Effect of temperature and pressure

Temperature as well as pressure, both influences the percentage of epoxy removal from the WPCB. Increase in both the temperature and pressure increased the percentage of epoxy removal. Sodium hydroxide being a stripping agent, it break down the strong epoxy bond in room temperature for a soaking time of 8 h to 24 h depending on the concentration of the solution. Temperature was increased from room temperature to desired temperature of 121 °C in autoclave; simultaneously the pressure was increased gradually. At this stage the water transforms into steam which helps in etching the epoxy surface of the WPCB soaked in chemical solution. For soaking under static condition, the 100% epoxy removal was achieved at 5 N of sodium hydroxide solution for a soaking time of 8 h along with some mechanical energy by Senophiyah-Mary et al., (2018). Instead of soaking under static condition, treatment under autoclave condition with enhanced temperature and pressure, the removal efficiency was almost similar and it was 100% even for solutions of lower concentration 0.25 N for 1 h using Autoclave. After treated under Autoclave, slight physical energy for scrubbing was needed to remove the weak epoxy adhered with the sample. On developing this methodology to a large scale industry, scrubbing over large scale was possible with the help of mechanically operated brush which would remove the weak epoxy adhered to the sample.

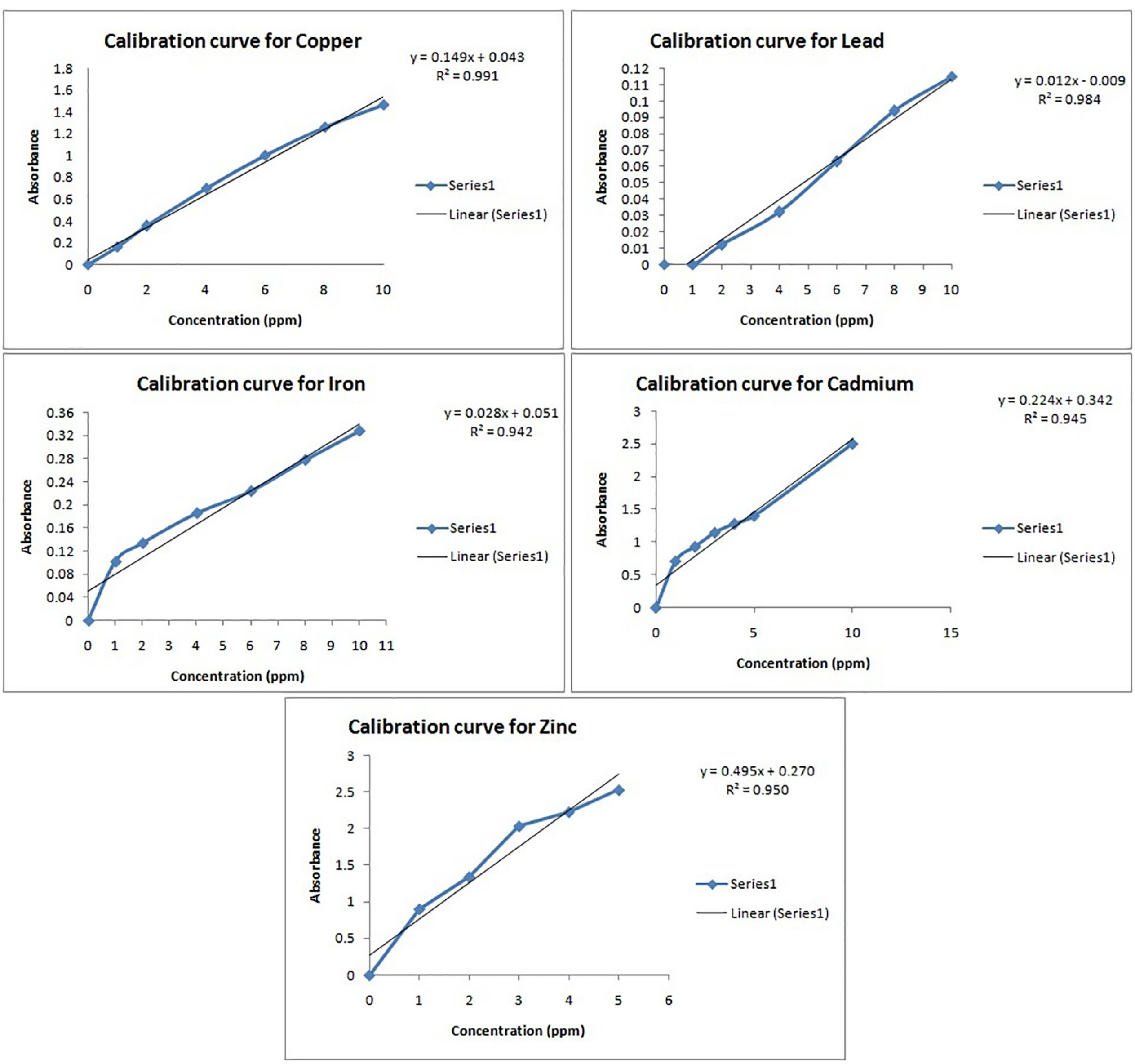

Fig. 6. Calibration curve for different metals identified using AAS.

3.3. Effect of volume of autoclave

Experimental procedure was completed likewise in autoclave of 108 L capacity with a temperature of 121 ˚C and a pressure of 1.1 kg/cm2 by keeping the WPCB samples individually and furthermore in bunch. Autoclave utilized in our current examination of capacity 55 L and 108 L had no separate temperature control, the temperature was set to 121 ˚C throughout the experimentation. When the desired pressure was reached, the steam valve ought to be opened for additional examination. Results in both the autoclave was similar and 100% removal efficiency was acquired regardless of whether the WPCB sample quantity was increased. Time required to reach the desired pressure in autoclave of capacity 108 L was 47 min and 45 min for individual and bunch of samples respectively.. From our examination, the volumetric limit of autoclave had no impact on epoxy removal efficiency; here the temperature set at 121 ˚C and the desired pressure played a significant part in stripping the epoxy layer.

3.4. Fourier Transform Infra-red spectroscopy analysis

Fourier Transform Infra-Red Spectroscopy was used to identify the functional group present in the samples. The stretch in the region of wave number 3300 corresponds to the vibrations in the OH bond. Samples were tested in FTIR for different concentrations of the prepared NaOH solutions one from lower concentration (0.25 N) and another from higher concentration (4 N) from our present study. Comparative FTIR analysis was presented for sample solution prepared as well as for the solution before and after filtration of the treated solution. FTIR spectroscopy shows similar peaks in all the three different circumstances(Figs. 4 and 5). Here the NaOH acquired the colour of the WPCB, for the solution of concentration less than 2 N and the peeled off epoxy is dispersed in the stripping agent. Settle down time was required for the particles to sediment at the bottom of the NaOH solution. The peaks obtained in the treated solution before and after filtration had the peak similar to the prepared sodium hydroxide solution which confirms that the epoxy was not being dissolved in the sodium hydroxide solution and the same sodium hydroxide solution can be reused further for removing the organic epoxy layer. Regeneration efficiency was about more than 95%, since the loss in regeneration was due to filtration process and the solution that wet the surface of the glass wares ().

3.5. Atomic Absorption spectroscopy (AAS)

Samples of different concentrations of NaOH solution such as 0.25 N and 4 N treated under autoclaving condition was analyzed for identifying the metal elements leached in it. Copper, Lead, Iron, Cadmium and Zinc was tested using Atomic Absorption Spectroscopy (TL-2800AA/TOPLAB INDIA) and the calibration curve was plotted against the concentration and absorbance of the stock solution. From the absorbance value of the sample, elements like Copper and Lead was identified and its leaching was confirmed in both the samples, whereas no trace of Iron, Cadmium and Zinc were identified in the NaOH solution (Fig. 6).

4. Conclusion

Several methods, techniques and solvents were used to remove the epoxy resin from WPCBs. Out of which only few were economically and environmentally feasible and consume less energy needed for the removal operation. NaOH solution of concentration 0.25 N under the autoclaving condition was a novel approach which was sufficient for complete removal of epoxy layer. After treated under autoclave, no soaking time was needed for WPCB in NaOH solution to achieve 100% removal efficiency. This novel approach using autoclave would consume less amount of NaOH which can be regenerated; regeneration was confirmed with the help of FTIR spectroscopy. Traces of copper (0.416 ppm & 0.013 ppm in sample 1 & 2) and lead (0.079 ppm in both samples) were identified using Atomic Absorption Spectroscopy.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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