



Separation of WPCBs by dissolution of brominated epoxy resins using DMSO and NMP: A comparative study



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HIGHLIGHTS

- NMP and DMSO were compared to separate WPCB layers to recover metals, glass fibers, polymers.
- Optimal cond. for NMP were temp. – 100 °C at 90 min., S/L ratio – 1:5, size/area – 4 mm/16 mm².
- Optimal cond. for DMSO were temp. – 90 °C at 90 min., S/L ratio – 1:2, size/area – 6 mm/36 mm².
- NMP and DMSO dissolved BER WPCBs; did not corrode metals and can be regenerated.
- Overall NMP proves to be better solvent than DMSO.

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ABSTRACT

Printed circuit boards (PCBs) in electrical and electronic equipment (EEE) abide of valuable and hazardous materials and due to its complex and discrete make up across manufacturers, processing of waste PCBs (WPCBs) is a massive challenge. And therefore either completely novel or improved processes are needed for recycling of WPCBs and recovery of valuable materials from it. Present comparative study, was performed for processing WPCBs using solvents *N*-methyl-2-pyrrolidone (NMP) and dimethyl sulfoxide (DMSO). Various parameters, which include WPCB sizes; solid to liquid (S/L) ratio; temperature and time, were investigated to understand the WPCBs processing by dissolving bromine epoxy resin using solvents. Results showed that the rate of removal and separation of the bromine epoxy resin (BER) increases with respect to increasing various parameters. Optimum condition of complete separation of WPCBs using NMP were S/L ratio of 1:5, WPCB size/area of 4 mm/16 mm² and 100 °C for 90 minutes, whereas for DMSO the optimum dissolution of bisphenol A were obtained in S/L ratio of 1:2, size/area of 6 mm/36 mm² at 90 °C for 90 min. Overall, NMP proves to be better solvent for bromine epoxy resin than DMSO in terms of bisphenol A dissolution and separation of various layers of WPCB. Used solvents can be vaporised–condensed under the decompression for regeneration. This novel process can be an eco-friendly and effective option for separation and recovery of various valuable materials such as metals, glass fibres, etc. from WPCBs. Further research and testing is needed for precise evaluation between two processes using NMP or DMSO solvents, in terms of the valuable material recovery from WPCB and process techno-economics.

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1. Introduction

Printed circuit boards (PCBs) are the essential, integral and predominant part of almost all electrical and electronic equipment (EEE). PCB provides the platform to mount various electronic items, such as resistors, relays capacitors, diodes, integrated circuits in a rigid manner. The rapid growth of technology [1] along with the high rate of obsolescence has led to an increase in E-waste

generation and consequently wastes PCBs (WPCB) [2,3] in developed and developing countries. Typically, PCB contains 50% polymer (epoxy resin or phenolic resin, along with brominated flame retardants (BFR), etc.), 20% glass fibre and 30% metals [20% Cu, 8% Fe, 4% Tn, 2% Ni, 2% Pb, 1% Zn, 0.2% Ag, 0.1% Au and 0.005% Pd] [3]. However composition of PCB wastes coming from different appliances, manufacturers and year of production is different. Though precious and valuable metals in PCBs are only 1% by weight, it accounts for 80% of the total intrinsic value [4] and thus gaining recycling attention from both formal and in-formal recyclers. Furthermore, PCB consists of multi-layers of etched metal

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conductive tracks laminated with fibreglass sheet, bonded with mixtures of polymer materials. Thermoset resin such as bromine epoxy resin (BER) is used as a flame retardant because PCBs are often exposed to high temperature both during manufacturing and operations [5]. BER are intermingled with metals [6] and glass fibres many times, which creates difficulty in individual components separation from WPCB and size reduction of WPCB for recycling.

PCB recycling involves two steps; (1) Mechanical–physical or metallurgical processing for dismantling and/or separation of different components and materials [7–10], (2) further separation and processing of metal streams; which probably is the most imperative step from economic and environmental viewpoint. Meanwhile, many studies, such as bio-metallurgy [11], combustion [12], hydrometallurgy [13], pyro-metallurgy [14], pyrolysis [15], supercritical fluid [16], and ionic liquid [17] were carried out for recycling of WPCBs. However these processes have their own limitations, the mechanical–physical processes are predominantly time and labour intensive. Whereas, the secondary metallurgical processes are energy and capital intensive as well as hazardous to environment as it may lead to formation of dioxins and furans, due to the presence of BFRs in PCBs [3]. While some processes may generate massive amount of waste acid, alkaline liquid and sludge, which may cause secondary pollution. This provides the necessity for the continued research on the recycling of WPCBs.

Kamo et al. [18] used tar derived from Japanese cedar and benzyl alcohol to liquefy the epoxy board at 250–300 °C under atmospheric pressure. Xing and Zhang, [19] employed a batch type reactor sub- and supercritical water to simultaneously degrade BER and recover metals from WPCBs. Ma et al. [20] and Guan [21], attempted to dissolve BER in PCBs using nitric acid; but the leaching rates were too low. However, only Zhu et al. [22] reported use of organic non-aqueous solvent dimethyl sulfoxide (DMSO) to separate WPCBs.

The present comparative study uses non-aqueous solvents *N*-methyl-2-pyrrolidone (NMP) vs. dimethyl sulfoxide (DMSO) for BER dissolution from the PCBs.

Both NMP and DMSO belong to the class of colourless dipolar aprotic industrial solvent and have the potential to dissolve numerous organic and inorganic compounds without corroding the metal. Table 1 provides comparative data of the physical properties of DMSO and NMP.

DMSO is highly stable below 150 °C and maintaining DMSO at 150 °C for 24 h, results in an expected loss between 0.1% and 1.0%. It has been reported that only 3.7% volatile material is produced when DMSO is held for 72 h at its boiling point (189 °C) [22]. Also, NMP has lower volatility (i.e., vapour pressure = 0.190 Torr at 25 °C) and evaporation rate – 0.03 (BuOAc = 1), thus its use releases fewer organic emissions to the atmosphere; biodegrades easily and

has low toxicity to aquatic life [23]. Therefore it can be summarised and infer that both NMP and DMSO have high thermal stability and can be easily recovered and recycled many times if it ends up in waste streams [24–30]. Due to the excellent safety characteristics NMP and DMSO finds wide range of applications, notably as a cleaning agent for electronic components, in plastic and petro-chemical industries, as a reaction solvent for pharmaceuticals, in agricultural chemicals and as a solvent for cellulose dissolution. In this paper, the effect of heating, particle size and solvent ratio (S/L) are evaluated on the dissolution of BER from WPCB using NMP/DMSO. The problem related to extraction of encapsulated copper from PCBs has been solved by the liberation of metallic thin layers by organic swelling. This research work aimed to compare between the organic solvents NMP and DMSO for developing a novel technique for separating valuable materials such as Cu, glass fibre, etc. from WPCBs in order to prevent environmental pollution from WPCBs processing.

To the best of our knowledge, there has been no previous comparative study reported, investigating the separation of WPCBs using NMP and DMSO.

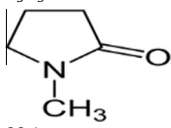
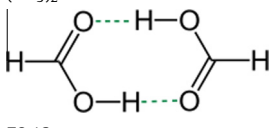
2. Materials and methods

2.1. Materials

WPCB of mother board extracted from desktop personal computer (PC) was used for this study. Discarded PC (manufactured in 2003) was collected from the stores' of NEERI (National Environmental Engineering Research Institute) Nagpur, India. The PCB of motherboard, as being the biggest in size and possessing more valuable materials in comparison with other PCBs found in desktop personal computers, was selected for this study.

Components such as ICs, transformers, fan, resistors, capacitors, pins, heat sink, motors, batteries mounted on the PCB were systematically and carefully detached manually using appropriate tools, to minimise occupational and environmental impact arising out of the various hazardous materials present in it. The bare WPCBs (after removal of mounted components from its surface) were cut into 3 cm × 3 cm size manually using plier/cutter and were further reduced to size using laboratory mixer grinder. The size reduced samples were separated according to particle size using an electromagnetic sieve shaker (Make: Electro Lab; Model: EMS8; Dimension: L339 × W312 × H270 mm). Mesh size of sieves used were 10, 8, 6, 4, 2, 1 and 0.71 mm. Sample was placed in the top sieve with the largest mesh and shaken in continuous and intermittent mode for 5 and 2 min respectively. The residue remaining on the sieves of mesh sizes 8, 6 and 4 mm of approximate size/area – 4 mm/16 mm²; 6 mm/36 mm² and 8 mm/64 mm² were used for conducting the experiments. All

Table 1
Physical property of NMP and DMSO.

Property	NMP	DMSO
Formula	C ₅ H ₉ NO	(CH ₃) ₂ SO
Structure		
Molecular weight (g/mol)	99.1	78.13
Boiling point (°C)	202	189
Molar volume	96.5	71.3
Viscosity (cP @ 25 °C)	1.67	2.0
Evaporation rate (BuOAc = 1)	0.03	0.026
Acute toxicity (g/kg rat oral)	3.6	14

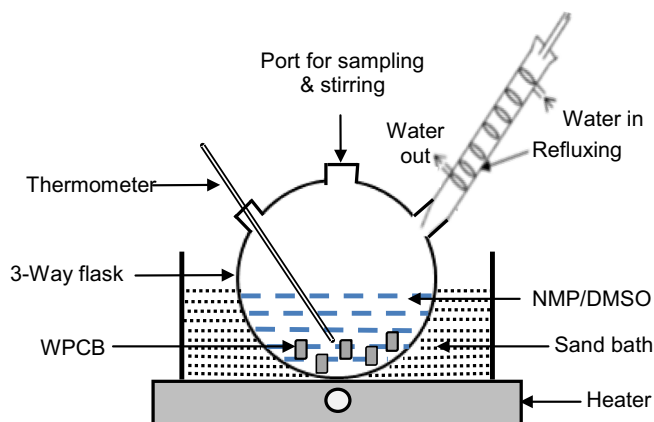


Fig. 1. Experimental set-up of the reactor for treating WPCBs in NMP/DMSO solvent.

reagents used in this experiment were analytical grade products from Fisher Scientific Pvt. Ltd., Mumbai, India.

2.2. Methods

The experiments were conducted using a 0.5 L, three-neck flask equipped with a continuous water circulating condenser unit. Fig. 1 shows the experimental set-up for separating the WPCBs. Reactor was charged with 5 g of WPCB sample each time along with appropriate solvent quantity. And once the desired temperature was reached, the set up was held constant at that temperature for 180 min. After every 30 minutes interval, a fixed quantity of solvent was withdrawn and the concentration was analysed in triplicate and only its mean values have been reported. The measurement errors were $\pm 1\%$. The experiments on WPCBs were carried out using NMP and DMSO with a varying experimental parameters such as WPCBs size/area (4 mm/16 mm², 6 mm/36 mm², 8 mm/64 mm²); temperature (70 °C, 80 °C, 90 °C, 100 °C) time period (30, 60, 90, 120, 150, 180 min); S/L (w/v) (1:2, 1:3; 1:4; 1:5) with random manual stirring.

The amount of bisphenol A, which is a component of BER [31], was measured to determine the BER concentration in NMP and DMSO. 0.10 g pure bisphenol A was transferred to a 100 ml volumetric flask dissolved and make up to volume with anhydrous ethanol to get stock solution of 1000 $\mu\text{g}/\text{ml}$. Concentration of 5, 10, 15, 20, 25, 30, 35, 40, 45 and 50 $\mu\text{g}/\text{mL}$ were prepared for making a standard curve (Fig. S1). The amount of bisphenol A was

determined using UV–vis spectrophotometer (AU-2701U, Systronics) with 1.0 cm matched quartz cells at a wavelength of 282 nm. To ascertain the accuracy of measurement the instrument was calibrated periodically using the standard procedure.

Rotary evaporation process was exercised to treat the used NMP/DMSO under decompression and the condenser was used to cool the vaporised gases at room temperature for solvent regeneration and residues left behind were collected and stored separately.

Fig. 2 shows a flowchart for dissolution process of BER regeneration of used NMP/DMSO solvent.

The residues i.e. treated WPCBs were washed with distilled water and dried at 85 °C for 6 h. Photographic images were taken of treated WPCBs. Transition in the chemical composition of used and unused; NMP or DMSO, were analysed using gas chromatography mass spectrometry, Perkin Elmer-Clarus 680 (GC) & Clarus 600-C (MS). The GC system was operated in split less injection mode. A 30 m DB-5 column (ID-320 μm) was used for component separation with the following temperature program: 150 °C for 10 min. Helium was used as the carrier gas at a constant flow rate of 1 mL/min. The GC injector temperature and MS ion source temperature were maintained at 150 °C. Sample extract (1 μL) standard solution was auto-sampled. The mass spectrometer was operated with electron ionisation (EI) mode with electron energy of 70 eV. Turbo mass software was used for the instrument parameters optimisation as well as for the data acquisition and analysis.

3. Results and discussion

The absorbency curves were plotted for the bisphenol A using the wavelength ranging from 200 nm to 300 nm. Bisphenol A shows maximum absorbance for UV light for the wavelength of 282 nm. Therefore, the concentration of BER dissolved into NMP or DMSO can be characterised by UV–vis spectrophotometer by determining the amount of bisphenol A.

Figs. 3–5 shows the effect of particle size/area, S/L ratio and temperature, respectively, on concentration of the BER dissolution in NMP and DMSO from WPCBs.

3.1. Dissolution mechanism

Organic swelling of WPCBs helps the solvents NMP/DMSO to penetrate into the WPCBs and dissolve the BER. Solvent penetrates the WPCB to soften the BER and gets absorb after certain duration. This causes the WPCB to swell and their mass to increase which leads to the dissolution of BER and subsequent separation of WPCBs layers, ultimately leading to the liberation of copper foil, glass fibre, etc. Moreover the hydrogen bonding plays an important role in the dissolution mechanism.

3.2. Using NMP as a solvent

Maximum bisphenol A concentration was seen for the 4 mm/16 mm² size fraction (Fig. 3) compared to the bigger size particles with smaller contact area. Since the interfacial surface area drives the overall mass transfer process, particles with small size or larger contact area results into higher dissolution rate of BER. It was observed that the concentrations of BER in NMP increase with an increase in S/L ratios (Fig. 4). The maximum concentration of bisphenol A is for the ratio 1:5 in comparison to 1:2 ratio and shows almost linear increase in concentration with respect to time. With the increase in temperature the concentration of BER dissolution increases linearly with respect to time (Fig. 5), may be due to fact that increase in temperature enhances the mass transfer.

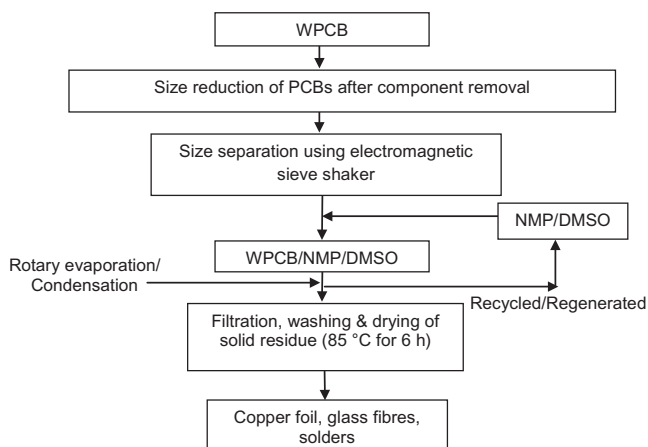


Fig. 2. Flowchart for dissolution of BER and regeneration of NMP/DMSO.

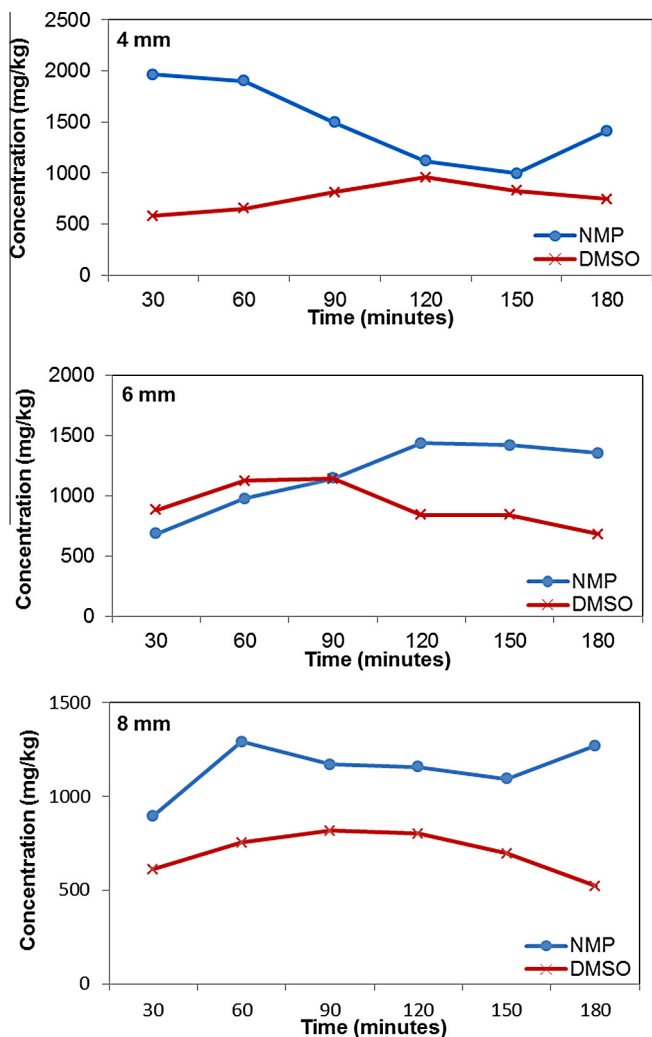


Fig. 3. Effect of size/area on concentration of the BER dissolution in NMP and DMSO from WPCBs.

3.3. Using DMSO as a solvent

The maximum bisphenol A concentration is seen for the 6 mm/36 mm² size fraction (Fig. 3). However the concentrations of BER in DMSO decrease with an increase in S/L ratio (Fig. 4). The maximum concentration of bisphenol A is for the ratio 1:2 in comparison to 1:5 ratio. Also with the increase in temperature the concentration of BER dissolution increases linearly with respect to time (Fig. 5). Almost all results obtained are in accordance with the results reported by Zhu et al. [32].

3.4. Solvent NMP vs. DMSO

Comparing and evaluating two organic solvents NMP and DMSO, for separation and removal of BER from WPCBs with respect to the effect of size/area, S/L ratio and temperature, it can be clearly ascertained that solvent NMP shows better results in terms of BER dissolution measured in terms of the bisphenol A concentration and in separation of BER layers. In all three cases with varying process parameters in terms of size/area (Fig. 3), S/L ratio (Fig. 4) and temperature (Fig. 5), it can be observed that the dissolution of BER in NMP is better than DMSO. Only for size fraction 6 mm/36 mm², S/L 1:2 and process temperature 90 °C DMSO shows better initial

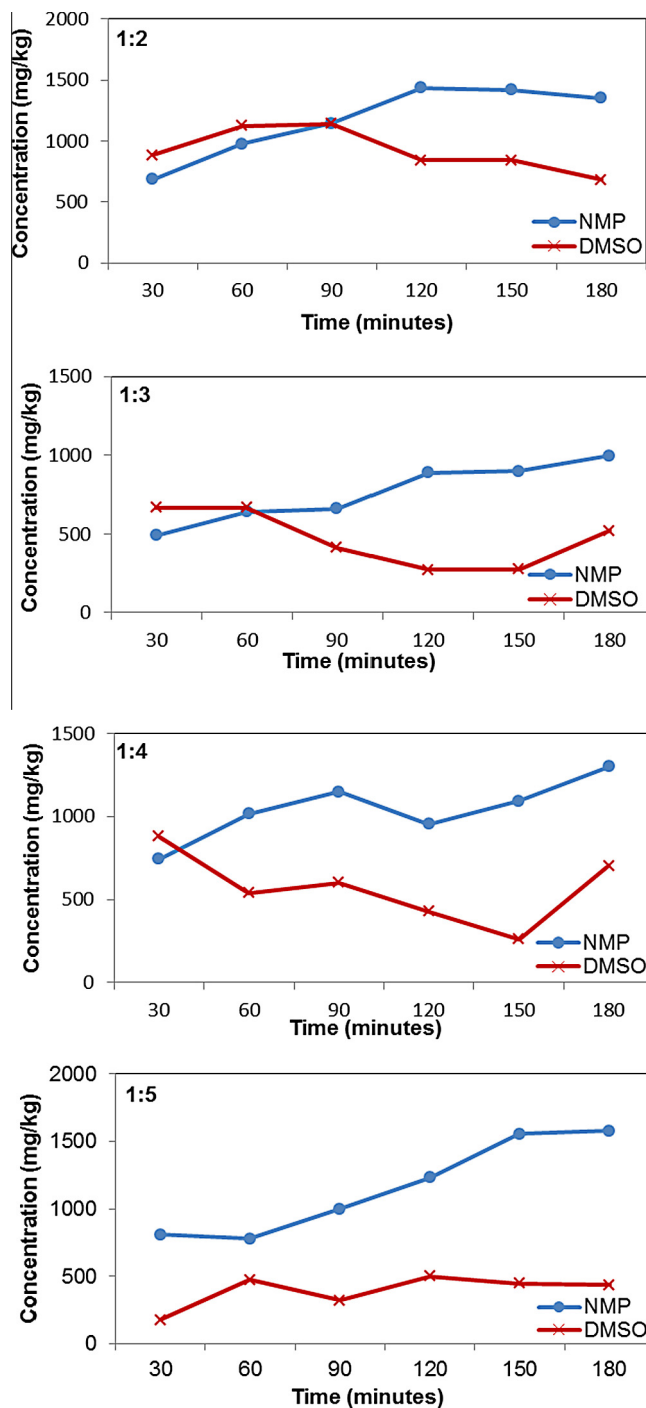


Fig. 4. Effect of S/L ratio on concentration of BER dissolution in NMP and DMSO from WPCBs.

results for first 90 minutes in comparison with NMP. But overall NMP substantiate to be a better solvent for removal and separation of BER for recovery of valuable materials from WPCBs. While solvent properties are significant in determining the separation performances [33], data are still lacking to fully understand the role of solvents in separation. Due to the complexity of solvent-polymer interactions in the reactions which differ in each type of polymer and solvent for a specific membrane type, it is difficult to accurately predict solubility behaviour [34]. Therefore it is presumed that the factors which may have influence the rate of

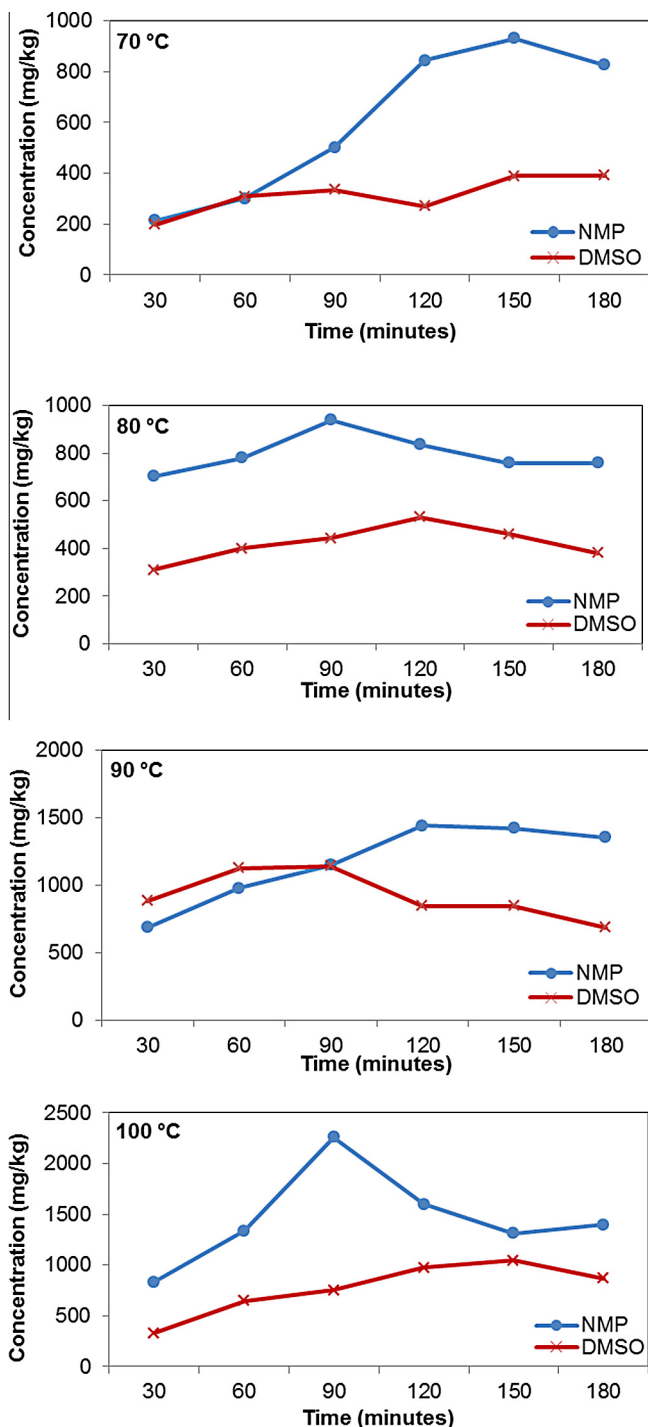


Fig. 5. Effect of temperature on concentration of the BER dissolution in NMP and DMSO from WPCBs.

dissolution and the reasons for NMP being a better solvent than DMSO may be due to its physical, chemical and structural properties, although both being polar aprotic organic solvents.

3.5. Physical properties

- *Solvent molar volume*: The penetration rate of any solvent depends on the molar volume of the solvent, higher the molar volume of the solvent better is the penetration rate which

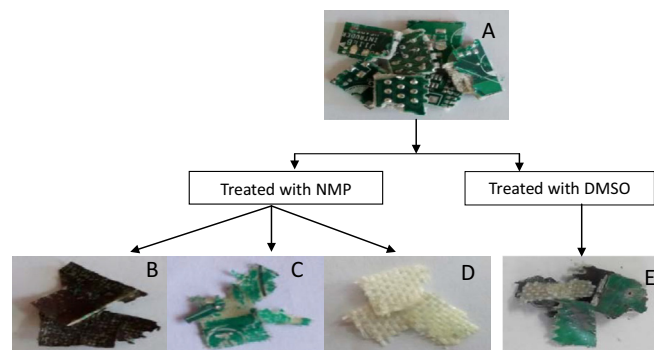


Fig. 6. Photographs of (A) untreated WPCB (B) copper foil and solder (C) liquid photo resist (D) glass fibre (E) DMSO treated WPCB.

results in better dissolution and vice versa. The molar volume of NMP is 96.5 much greater than that of DMSO which has 71.3 [35].

- *Viscosity of solvent*: High viscosity solvents have a lesser collision frequency per unit time thus they diffuse at a slower rate through any particles as compared to the less viscous solvent. Thereby the reaction rate of most of reaction decreases rapidly with increase in viscosity. Viscosity of DMSO is 2.0 cP @ 25 °C which is higher than that of NMP with 1.67 cP @ 25 °C.

3.6. Chemical and structural properties

- *Electronegativity (X)* i.e. capability of an atom or functional group to attract electrons is higher in NMP due to presence of 'N' atom ($X = 3.04$) in comparison with DMSO with 'S' atom ($X = 2.58$) and hence can form co-ordinate bond (dative covalent bond), by giving a pair of electron, with the BER. The shared electron pair will be pulled more strongly by the atom with the higher electronegativity i.e. 'N' atom of NMP. As a result, a polar covalent bond is formed.
- *Capability of NMP to form hydrogen bonding with BER* is more than that of DMSO due to the presence of 'N' atom.

Finally, the metals (copper foils), glass fibres and liquid photo solder resists are distinctly detached from one another and can be easily separated using NMP as solvent. Also various techniques can be applied for its separation, based on the density difference principal.

Fig. 6A–E depicts the photographs of WPCBs treated under the condition of S/L ratio of 1:5, WPCBs area of 6 mm/36 mm², 180 min using NMP and DMSO. It can be observed from Fig. 6 that the copper foils, liquid photo resists and glass fibres were clearly separated from the WPCBs treated with NMP solvent whereas in case of DMSO it is still intact. The solders were still bonded to the surfaces of the copper foils in both cases.

Furthermore, the liquid photo solder resists were detached from the copper foils (Fig. 6B and C). The copper foil does not show any mark of corrosion which also substantiate the non-corroding property of NMP and DMSO towards metal. Rotary evaporation process was exercised to treat the used NMP/DMSO and the condenser was used to cool the vaporised gases for NMP/DMSO regeneration. Subsequent colour change of the unused solvent from initial colourless to final greenish appearance, further ascertained the dissolution of BER in both the solvents (Fig. S2).

Figs. 7 and 8 shows the GC–MS chromatogram and fragmentation patterns of the used and unused NMP and DMSO, respectively, which clearly reflects the identical characteristic peaks and patterns of the NMP and DMSO, which indicates that the chemical properties of the used solvent has not change with reference to

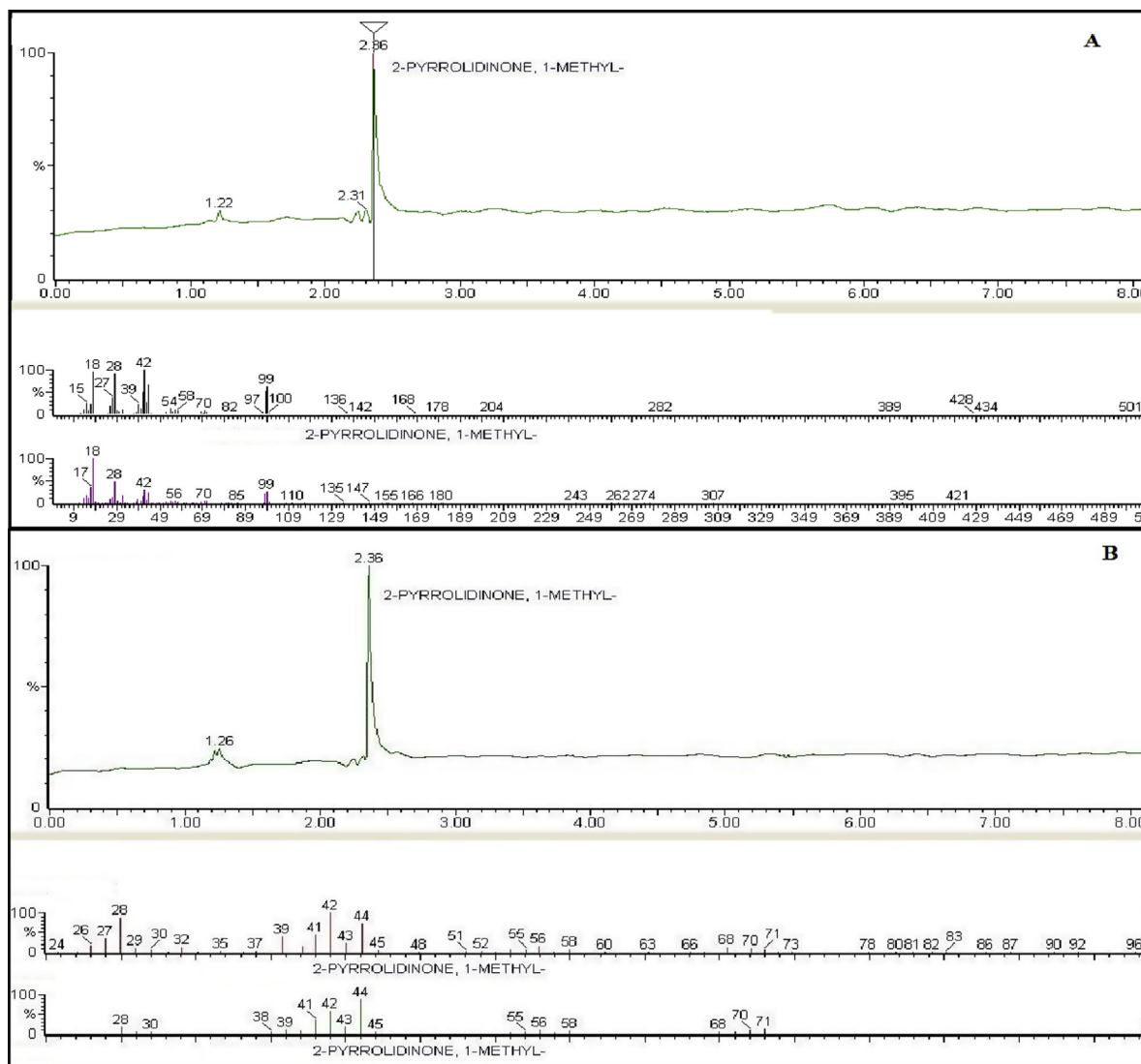


Fig. 7. GC-MS chromatogram of (A) unused and (B) used NMP solvent.

unused solvent. From the perspective of protecting the environment and decreasing costs, it is noteworthy that NMP and DMSO can be regenerated.

As both the polar solvents NMP and DMSO are good hydrogen bond acceptor, that is results in the formation of various possible hydrogen bonds between the BER and solvents, which may be responsible for the separation of BER.

In general, separation of WPCBs using NMP may have explained that NMP easily allows the formation of hydrogen bonds between the OH of BER and N-O group of NMP (Fig. 9).

Fig. 9A shows the hydrogen bond of $H \cdots O$. The hydrogen from the OH group in the BER pulls electrons from the oxygen in the NMP. Fig. 9B shows that the hydrogen in the methyl group of the NMP forms hydrogen bonds with bromine and oxygen in BER. Fig. 9C shows the hydrogen bonds of $H \cdots N$. The strongly electronegative nitrogen in the NMP pulls the hydrogen in the OH group of the BER.

Whereas separation of WPCBs using DMSO is due to the formation of hydrogen bonds between the OH and Br of BER and O-S group of DMSO; the bromine in the BER pulls electrons towards itself. Meanwhile, hydrogen in the methyl group in DMSO may form hydrogen bonds with bromine and oxygen in BER (Fig.10) [32].

Hydrogen bond of $H \cdots O \cdots Br$ is shown in Fig. 10A. The electron is attracted towards oxygen in the DMSO due to sulphur, which accepts the hydrogen from the hydroxyl groups in the BER. Meanwhile, the electrons also get pulled by the bromine in the BER towards itself. The hydrogen bonds of $H \cdots O \cdots S \cdots O$ is shown in Fig. 10B. The oxygen in the BER pulls electrons towards the sulphur of the DMSO, which are transfer to the oxygen of DMSO for accepting the hydrogen from the hydroxyl in the BER. Fig. 10C shows that the methyl group of DMSO gives its hydrogen to form hydrogen bonds with oxygen and bromine in BER [32].

However, there is a need for detail study and research to understand the exact mechanism of NMP and DMSO dissolving BER in WPCBs.

4. Conclusion

Organic solvents such as NMP and DMSO can be utilised for processing the WPCBs of a typical electronic waste. These solvents can be used to completely separate the various layers of the WPCBs, which enable easy recovery of valuable materials including metals, glass fibres, etc. The optimum condition of complete separation of WPCBs using NMP solvent was S/L ratio of 1:5, WPCB size/area of 4 mm/16 mm² and 100 °C for 90 min, whereas for the DMSO the

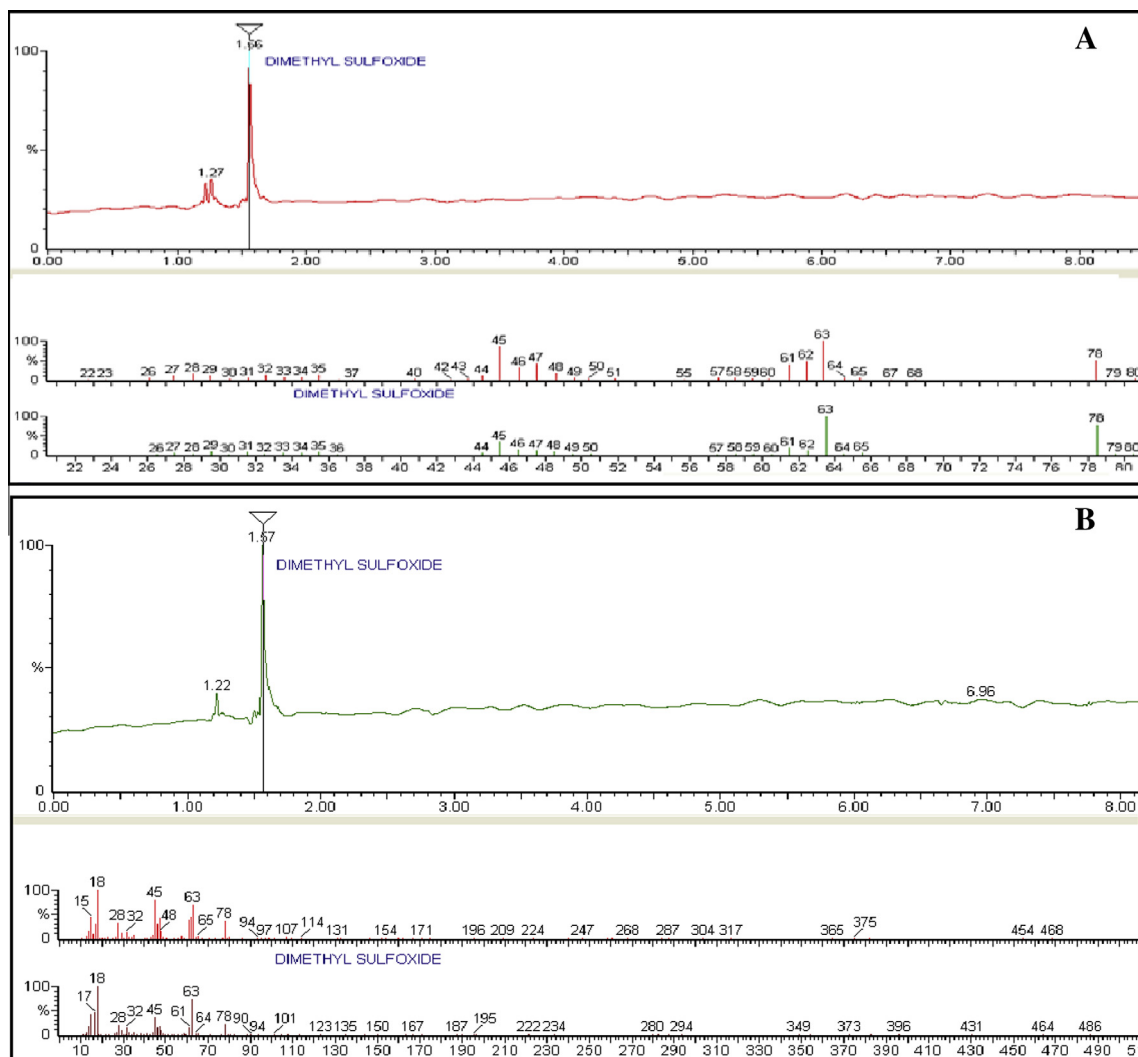


Fig. 8. GC-MS chromatogram of (A) unused and (B) used DMSO solvent.

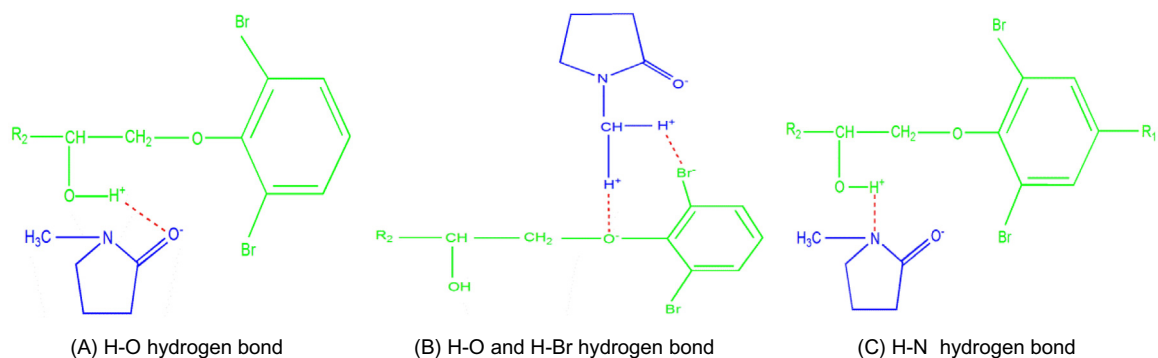


Fig. 9. Potential role of hydrogen bonding between NMP and BER.

optimum dissolution of bisphenol A were obtained in S/L ratio of 1:2, size/area 6 mm/36 mm² and 90 °C for 90 min. Overall NMP proves to be better solvent for BER in comparison to DMSO in terms of bisphenol A dissolution and separation of various layers of epoxy resin. Rotary evaporation process was exercised to treat the used NMP/DMSO and the condenser was used to cool the

vaporised gases for NMP/DMSO regeneration. NMP and DMSO not only dissolve the BER of WPCBs but also did not react with metals, liquid photo solder resists, and glass fibres. This new process using organic solvent does not produce bromine volatilise responsible for causing secondary environmental pollution. This complies with the principle of sustainable development by

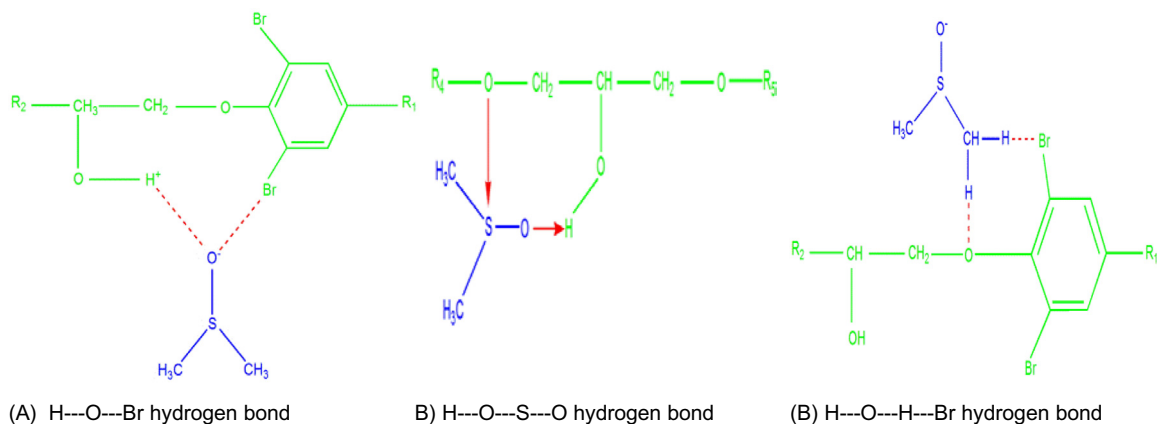


Fig. 10. Potential role of hydrogen bonding between DMSO and BER.

decreasing the manufacturing cost of treating WPCBs to achieve complete separation and recovery of reusable material. Further research and testing is needed for precise evaluation and grading merit between two processes using solvent either NMP or DMSO, in terms of valuable material recovery from WPCB and process techno-economics.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.cej.2015.06.007>.

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