



Enhanced extraction of brominated flame retardants from e-waste plastics

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ABSTRACT

Acrylonitrile butadiene styrene (ABS) is a high utility engineering thermoplastic, abundantly present in waste electronic and electrical goods. The demand for this plastic resin increases continuously with rapid industrial growth across the world. During the recycling of e-waste plastics, it is a common occurrence to find brominated flame retardants, such as Tetrabromobisphenol A (TBBPA) in the plastic. These flame retardants are intentionally added to the plastics as additives. Three processes were investigated to find the optimum solvent/antisolvent combinations, process parameters for the efficient recovery of TBBPA free plastics: microwave assisted extraction (MAE), ultrasound assisted extraction (UAE), and dissolution reprecipitation (DR). Rigorous solvent screening was carried out for both TBBPA and ABS using Hansen Solubility Parameters (HSP), and their interactions with the solvents were investigated with a molecular based Conductor like Screening Model for Realistic Solvents (COSMO-RS) software platform. A 4:1 (v/v) solvent mixture of 1-propanol and heptane was found to be ideal for the solid–liquid extraction process. The optimal extraction conditions (Temperature = 110–130 °C, time = 60 to 70 min solid/liquid ratio of 0.025) in MAE were predicted using machine learning based Support Vector Regression (SVR) technique. MAE was found to be superior to UAE and DR processes in terms of quantity and quality of recovered plastics. The maximum extraction efficiencies for TBBPA removal were achieved with MAE, UAE, and DR processes at 95.81%, 93.92%, and 98.15%, respectively. However, the DR process had a relatively lower plastic recovery.

1. Introduction

Brominated flame retardants, commonly known as BFRs, refer to a group of chemical additives that contain organobromine. These additives are added to materials like plastics, synthetic resins, textiles, electronic circuit boards, and furniture to reduce their flammability and prevent the rapid spread of fire [1]. Tetrabromobisphenol-A (TBBPA) is a common BFR found in the plastics from waste electronic and electrical equipment (WEEE). These compounds are heat and light sensitive, as well as persistent and capable of bioaccumulation [2–4]. The adverse health and livelihood effects of various BFRs can be found elsewhere [5,6]. As per the Toxic Substances Control Act (TSCA), the U.S. Environment Protection Agency (EPA) has listed TBBPA as a chemical for risk evaluation[7]. The Occupational Safety and Health Administration (OSHA) has not defined an occupational exposure limit for TBBPA but

has recommended a permissible exposure limit for Particulate Not Otherwise Regulated (PNOR) during industrial operations [8]. Due to its extensive usage in consumer goods, the chances of human exposure of TBBPA are very high. TBBPA and its derivatives were identified as priority substances for screening assessment under the Canadian Environmental Protection Act, 1999 due to their persistence and inherent toxicity to non-human organisms [9]. The EU directives (2012/19/EU) on managing WEEE suggested selective treatment for the plastics containing BFR[10]. WEEE plastics are subject to regulations which limit the presence of BFRs to 2,000 parts per million (ppm) of bromine. This is because available plastic sorting technologies are unable to differentiate between restricted and non-restricted BFRs. An operational threshold of 2,000 ppm total bromine has been set to aid in the separation of plastics containing BFRs[11]. Different BFRs and its derivatives have been found in freshwater fish[12], household dust laundry wastewater[13,14],

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indoor air[6], agricultural products[15], toys[16], etc. Other studies also confirmed the presence of the bioaccumulative BFRs in different marine animals, such as predator fish [17], trout, salmon[18], bivalve [19], and sharks [20]. Thermal treatments, such as incineration or pyrolysis of BFR-laden plastics, can create conditions that are favorable for the generation of highly toxic polybrominated dibenz-p-dioxins (PBDD) and polybrominated dibenz-furans (PBDF)[21,22]. These compounds are highly toxic and considered as persistent pollutant, bioaccumulative[23] and can cause range of adverse health effect [24]. Other studies highlighted that these compounds affect the reproductive hormones and thyroids of laboratory animals[20,25–27].

Heat plays a vital role in various production processes, including injection molding, extrusion, 3D printing, and others. However, the presence of BFR in the plastic during heating can result in the emission of toxic fumes, posing a serious health hazard to individuals in the vicinity[28]. Furthermore, dumping of such plastics can lead to the leaching of brominated components into the environment [29,30]. Sun, et al. [31] have argued that there is even a subtle diffusion of the BFRs from the plastic to the air through molecular matrix, and this diffusion can be significantly enhanced by abrasion tear and pulverisation of the plastics. Hence, it is essential to remove the excess BFR from the plastics before recycling.

Sun, et al. [31], Wan, et al. [32] reported the mass transfer kinetics involved in the release of BFRs such as PBDEs, TBBPA-bis-(2,3-dibromo-2-methylpropyl ether) from plastics, respectively. Which implies that the interaction between the polymer, the additive, and the solvent is an important consideration in the solvent extraction of additives from plastics [33]. For efficient extraction, the solvent must diffuse into the polymer matrix by swelling/softening the polymer, followed by dissolution of the TBBPA (BFR), and diffusion back into the solvent phase with minimal loss to the polymer [34,35].

In this study, solvent-based green recycling of ABS impregnated with TBBPA from e-waste stream was performed using suitable solvents and solvent mixtures by extraction. The solvents were screened using Hansen Solubility Parameter (HSP) calculation to achieve optimal extraction efficiency. The recovery of pure polymers from contaminated plastics is important to reduce the production of virgin plastics from petroleum sources, hence decrease the carbon footprint and also fulfill the circular economic goal [36]. Statistically, the recycling of such plastics is significantly low due to process complexity, polymer contamination, and limited profitability. Prior to 2020, only 10% of all plastic waste generated had been recycled, the rest was either incinerated or discarded [37]. Hence, a sustainable and economical recycling of plastic will greatly reduce CO₂ emissions, plastic accumulation in landfills, and environmental hazards associated with plastic. It was estimated that 30% of e-waste plastics are ABS [38,39]. ABS Plastics are made from acrylonitrile, butadiene, and styrene by emulsion polymerization. Acrylonitrile is a synthetic monomer made from propylene and ammonia. Butadiene is a product of hydrocracking petroleum, and styrene is produced by dehydrogenation of ethylbenzene. All these processes are highly energy intensive and carbon negative. ABS is one of the most important plastic components, used primarily in electronics, electrical equipment, and automotive parts due to its high impact strength and flexibility. The global production of ABS plastic reached 11.38 million metric tons in 2020 [40]. The cost of ABS plastic has risen sharply recently, reaching \$3,305/ton (injection molding grade) in the USA[41]. Recycling of pristine ABS from e-waste will increase the availability of these plastics.

Earlier studies have identified the problem associated with BFR laden plastics and proposed various methods, such as extraction of the BFR [42–44], thermal degradation of plastic by pyrolysis for the transfer of Br into residue products[45,46] and for direct safe reuse as recycled products[47]. Evangelopoulos, et al. [48] reported reduction of Br in the order of 10 – 37% from Bromine-containing plastics using isopropanol and toluene as solvents under Soxhlet extractor, albeit no findings on recovery of plastics were reported. Vilaplana, et al. [42] reported TBBPA

removal efficiencies of 90.05% and 67.64% from high impact polystyrene (HIPS) in microwave assisted extraction (MAE) using an isopropanol/n-hexane (1:1 v/v) mixture and an isopropanol/methanol (1:1 v/v) mixture as solvent, respectively. Bhaskar, et al. [49] carried out the microwave irradiation of BFR laden HIPS using aqueous organic solvents with triethylene glycol and achieved a Br removal efficiency of 85 % (w/w) at 250 °C and 30 min run time. Past studies concentrated on the separation of the BFR from the plastic, but little attention was paid to the quality of the recovered plastic. A detailed mechanism of the solvent and polymer/BFR interaction was lacking. Hence, this study focuses on the screening of appropriate solvents to improve extraction using microwaves or ultrasound and also investigates the plausibility of the dissolution and re-precipitation method. Here, we have reported a computationally guided approach to rationally select solvents using Hansen solubility parameters (HSPs), and demonstrated the mechanism of interaction between the solvent and the polymer/BFR [50]. This was performed using the platform combining quantum chemical and statistical software called “conductor-like screening model for realistic solvents” (COSMO-RS). Solvent properties, including solubility and polarity, significantly impact the recovery of plastics and the extraction of specific additives during solvent extraction processes. These properties are crucial for determining the effectiveness of the extraction method. The optimal process conditions were determined using the machine learning based Support Vector Regression (SVR) technique. The post-process recovered polymers were analysed intensively and comparisons were made for all the process outcomes in terms of efficiency and polymer recovery. The findings of this study will significantly contribute to the e-waste (plastic) recycling industry.

2. Materials and methods

2.1. Sample selection and preparation

Three real ABS plastic samples from real e-waste sources and a customized sample (CS) with a known concentration of BFR (Tetrabromobisphenol-A) were chosen for this study (Table 1). The preparation process of the real sample is explained in [Text 1S in the supporting information](#) file. The CS in granular form was obtained from Shandong Tianyi Chemical Company, China. The standard TBBPA, pure ABS as 3D printing filament (ABS3D), and all the organic solvents (Purity > 99.5%) for the screening were procured from Sigma Aldrich Singapore. The Br concentration was measured by X-ray fluorescence spectroscopy ([Text 2S](#)).

2.2. Calculation of the solubility score (S_i) for solvent screening

0.1 g of the solid samples (ABS3D, TBBPA) was mixed with 10 ml of the solvent at 40 °C and kept under ultrasound (Qsonica sonicator, Q125, 125 W, 20 kHz) for 30 min. Solubility was observed visually ([Fig. 2S](#)) and assigned solubility scores (S_i) of 0, 1, and 2 for no dissolution, partial dissolution, and fully dissolved solutions, respectively. The solubility score was used for the calculation of the Hansen solubility parameters (HSP). The HSP calculation method is explained in the [supporting information](#) ([Text 3S](#)).

Table 1
BFR laden e-waste plastic samples.

Sample ID	Source	Br concentration (%w/w)
ReBRS1	Wireless router (black)	9.77 ± 0.27
ReBRS2	Wireless router (white)	12.4 ± 0.38
ReBRS3	Paper shredder (white)	9.5 ± 0.25
CS	Customized sample	8.3 ± 0.97

2.3. Extraction processes

Following the solvent screening, microwave assisted extraction (MAE) was performed, and the parameters were optimized for maximum efficiency. Initial trial experiments were conducted at a temperature (T) of 110 °C; for time (t) up to 90 mins; and a solid to liquid ratio (s/l) of 0.025 g/ml based on past studies [51]. The Br extraction efficiency (%) and the recovered plastics (%) were calculated based on the Eq. (1) and Eq. (2), respectively. The parameter limits are listed in Table 2. Parameters were selected based on preliminary experiments, and the maximum temperature was limited to 130 °C, considering energy efficiency and thermal stability of the polymer. The parameter optimization was carried out using support vector regression (SVR) based machine learning (ML) algorithm. Details of the MAE process can be found in Text 4S in the supporting information file.

$$\text{Br extraction efficiency}(\%) = \frac{(\text{Br concentration})_{\text{sample}} - (\text{Br concentration})_{\text{recovered plastics}}}{(\text{Br concentration})_{\text{sample}}} \times 100 \quad (1)$$

$$\text{Recovered plastics}(\%) = \frac{\text{Weight of the recovered plastics}}{\text{Weight of the untreated sample}} \times 100 \quad (2)$$

For comparative understanding, ultrasound-assisted extraction (UAE) was performed along with the dissolution and reprecipitation (DR) method. Both the processes have been explained in detailed in the Text 5S and Text 6S in the supporting information file. Schematic representation of all three-extraction setup is shown in Fig. 1.

Analytical methods such as X-ray fluorescence spectroscopy (XRF), Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR), Differential scanning calorimetry (DSC), Thermogravimetric analysis (TGA) and Gas chromatography and mass spectroscopy (GCMS) used for the sample analysis are explained in the supporting information (Text 7S).

3. Results and discussion

3.1. Solvent screening

3.1.1. Solvent screening using HSP

ABS is a high-impact thermoplastic composite, which is chemically stable but sensitive to organic solvents like ketones, aromatic and chlorinated hydrocarbons [52]. On the other hand, BFRs are organic chemical additives soluble in most of the organic solvents. To achieve maximum extraction efficiency, the solvents must be able to dissolve the BFRs without dissolving the polymer. From HSP calculation, the ideal solvent or solvent mixture for the extraction of BFR from ABS plastic was identified. The HSP sphere of ABS and TBBPA along with the solvent's positions are shown in Fig. 2. The HSP values along with Relative energy difference (RED) of the solvents/solvent mixtures can be found in

Table 2
Parameter limit for the DOE.

Parameter	Symbol	Range min	max
Temperature (°C)	T	30	130
Time (min)	t	10	90
solid/liquid ratio (g/ml)	s/l	0.025	0.1

Table 1S. The calculated values of HSP for TBBPA and ABS are listed in Table 3S. For TBBPA all solvents except water, dodecane, cyclohexane, n-hexane and heptane (Hp) were proved to be good solvents (RED < 1). Solvents like DMF, NMP, acetonitrile, DCM, acetone, ethyl acetate, tetrahydrofuran (THF), toluene, 2-butanone are able to dissolve ABS plastic (RED < 1). Alcoholic solvents (and water) appeared to have a low affinity toward dissolving the ABS (RED > 1). Investigation with the help of the COSMO-RS[50] gave us added insight into the interaction of the solvent with the polymer and the BFR. The alcohol solvents are considered polar solvents and effective towards TBBPA dissolution. Control amounts of heptane (Hp) (non-polar solvent) were mixed with 1-propanol (1P) in a volume ratio of 1:1, 4:1, and 1:4 to increase the effectiveness of the alcohol solvent for the extraction of TBBPA from the ABS plastic matrix. The screened solvents are safe and economical for industrial application [53]. For the DR process, THF was chosen as a

good solvent to completely dissolve the plastics for DR process. The RED values of THF for both ABS and TBBPA are at 0.68 and 0.43, respectively (Table 1S). 1P is chosen as non-solvent to precipitate the ABS and keep TBBPA dissolve in the liquid phase.

3.1.2. Cosmo-Rs calculation

The calculation process in the COSMO-RS is explained in detail in the supporting information (Text 8S and 9S). The framework of COSMO-RS and the simulation flow representing the TBBPA molecule and the isolated ABS molecule with σ -surface are shown in Fig. 3(a). The corresponding σ -profile and σ -potential/ $\mu(\sigma)$ with respect to the ABS, TBBPA and solvents are shown in Fig. 3(b) and (c), respectively.

The σ -profiles reflect the polarity comparison between the solute and the solvent. There are three regions in the sigma profile: the values of σ between -0.01 to $+0.01 \text{ e}/\text{\AA}^2$, $-\infty$ to $-0.01 \text{ e}/\text{\AA}^2$ and $+0.01$ to $+\infty/\text{\AA}^2$, as the nonpolar region, hydrogen bond (H-bond) donor region and H-bond acceptor region, respectively[54]. The σ -profile peaks' intensities beyond $\sigma = -0.01 \text{ e}/\text{\AA}^2$ and $+0.01 \text{ e}/\text{\AA}^2$ correspond to the strength of the H-bond donor and the acceptor region on the surface. A larger peak beyond $\pm 0.01 \text{ e}/\text{\AA}^2$ increases the probability of H-bond interaction, and the further the peak is from the center, the stronger the H-bond interaction. The peaks of the nonpolar segments of the surfaces are visible in the range near $\sigma = 0 \text{ e}/\text{\AA}^2$. An asymmetric σ -profile can be seen for ABS and TBBPA (Fig. 3(b)). Indeed, in the case of ABS, a narrow peak beyond -0.01 and $-0.02 \text{ e}/\text{\AA}^2$ of the σ -region and the peak shifting slightly away from the center generally corresponds to the presence of H-bond donor sites. However, a slightly broad peak at $0.01 \text{ e}/\text{\AA}^2$ represents the influence of π -electrons of the $-C \equiv N$ group of the ABS molecule, represented by the slightly red colored σ -region (H-bond acceptor) (Fig. 3(a)). The σ -potential ($\mu(\sigma)$) of the ABS shows a distinct shape (Fig. 3(c)). The negative side of the σ -region ($\sigma > -0.01 \text{ e}/\text{\AA}^2$) has more negative value, indicating its affinity to interact with H-bond donor surfaces. On the other hand, the positive value of $\mu(\sigma)$ in the region ($\sigma > +0.01 \text{ e}/\text{\AA}^2$) reflects that ABS lacks the H-bond donor surface and has the affinity to attract H-bond acceptor surfaces. The σ -profile of the TBBPA is dominating in the non-polar region ($\sigma \sim 0 \text{ e}/\text{\AA}^2$) with a narrow cusp near $0.01 \text{ e}/\text{\AA}^2$ representing the π -electrons of the ring, and a small bump in the region of the $\sigma = -0.01/\text{\AA}^2$ representing the electronegativity around the oxygen atom. However, the $\mu(\sigma)$ values of TBBPA have positive values in the region of $\sigma > -0.01 \text{ e}/\text{\AA}^2$ and negative values in a wider range in the region $\sigma > +0.01 \text{ e}/\text{\AA}^2$, which signifies the availability of H-bond acceptor surfaces on the molecule. At the

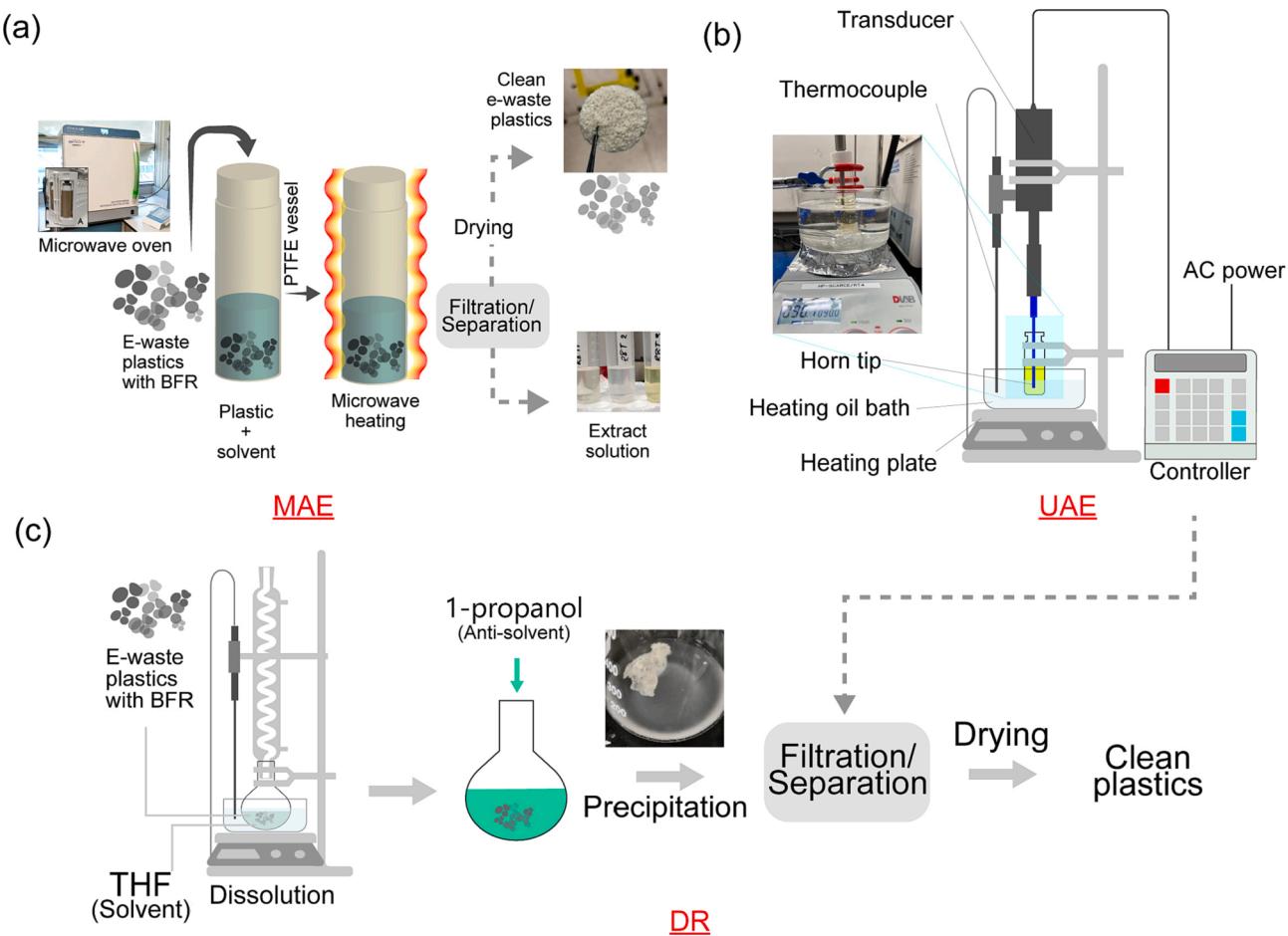


Fig. 1. Schematic representation/experimental setup of (a) MAE, (b) UAE and (c) DR process.

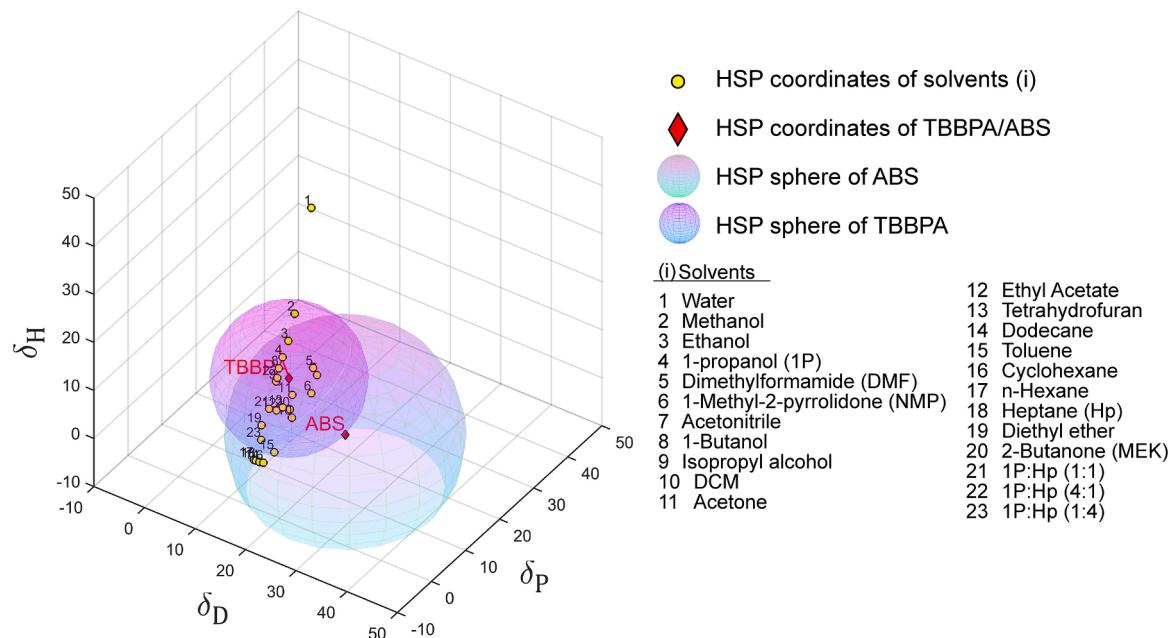


Fig. 2. 3D HSP sphere of ABS and TBBPA along with the solvent coordinates.

molecular level, both ABS and TBBPA have weak H-bond donor and H-bond acceptor surfaces, and strong peaks in the region near the non-polar value ($\sigma = 0 \text{ e}/\text{\AA}^2$) of the sigma profile. Both the molecules of

ABS and TBBPA do not participate in polar interaction and the most favorable interactions are the CH- π and π - π interaction. Solvents such as heptane, which do not show any polarity, have a σ -profile with a broad

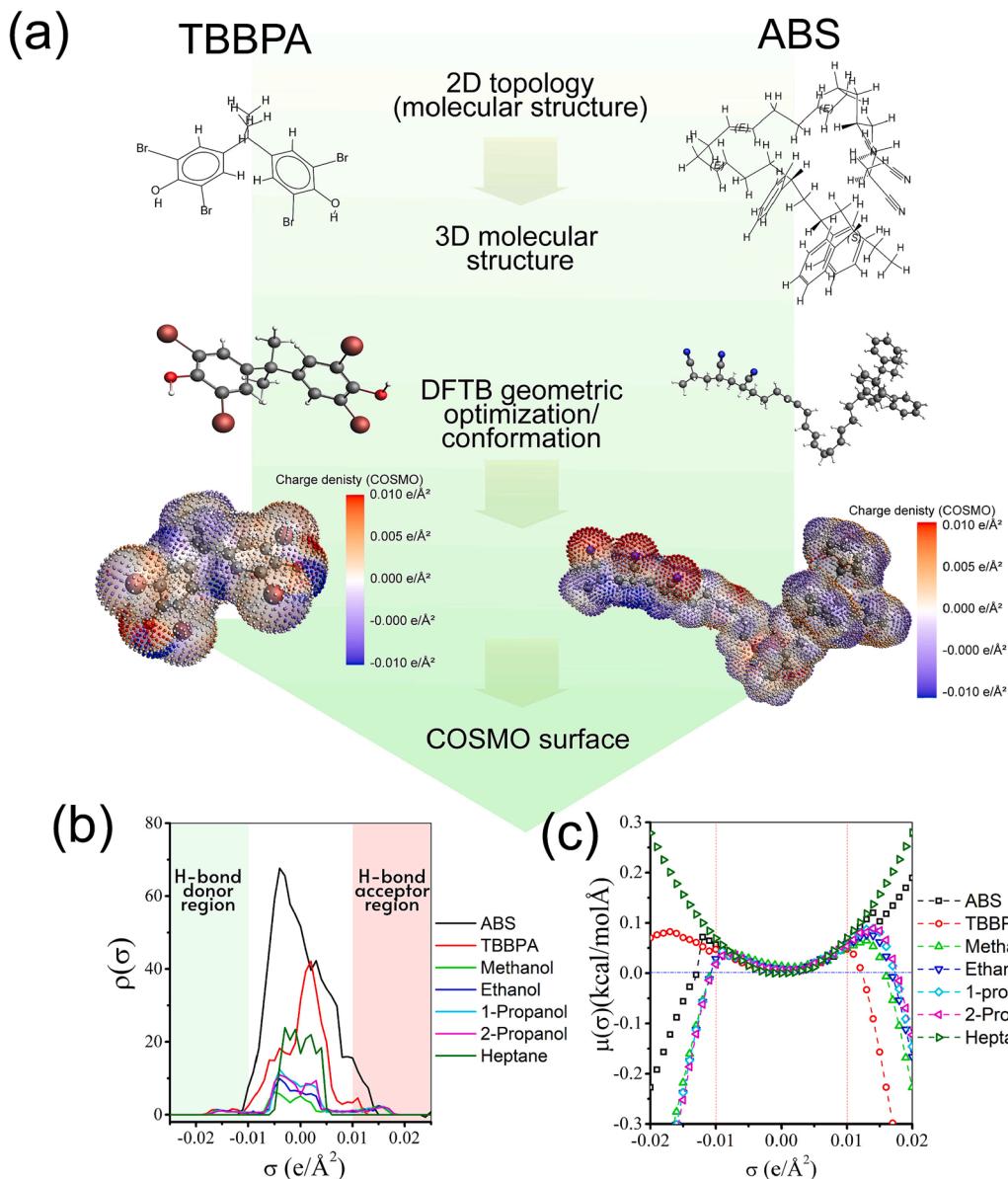


Fig. 3. (a) Chemical structure and COSMO cavities (charge distribution) of isolated ABS and TBBPA molecules with the simulation flow, (b) σ -profile and (c) $\mu(\sigma)$ of ABS, TBBPA and selected solvents predicted by COSMO-RS.

peak distributed near the $\sigma = 0 \text{ e}/\text{\AA}^2$ region and a concave upward parabolic $\mu(\sigma)$. On the other hand, the σ -profiles of the alcohol solvents have strong central peaks from the alkane chain along with an inferior peak in the region of $\sigma = -0.01 \text{ e}/\text{\AA}^2$ and $0.01 \text{ e}/\text{\AA}^2$ resulting from the marginally positively charged surfaces near the oxygen and the polar hydrogen in the molecules. The σ -profile and $\mu(\sigma)$ of the solvent mixtures (1P and Hp) attributes the dominant properties of both solvents (Fig. 5S) and the shape of the resulting $\mu(\sigma)$ is more like 1P, with a downward decline in both the directions of $\sigma = \pm 0.01 \text{ e}/\text{\AA}^2$, corresponds to its affinity for both H-bond donors and acceptor surfaces [55].

3.2. Extraction of TBBPA using MAE, UAE, and DR process

The microwave heat is transferred to the sample via a dual mechanism of dipole rotation of the solvent and the sample, as well as ionic conduction [35,56,57]. Experimental outcomes of the MAE are shown in Fig. 4 for the solvent mixture 1P / Hp performed at initial experimental conditions. Fig. 4(a) and 4(b) show the % Br extraction efficiency and the plastic recovery for three mixing ratios and for all four samples,

respectively. Using a 4:1 mixing ratio of 1P to Hp solvents, the maximum extraction rates were achieved for all four samples, as depicted in Fig. 4(a). Specifically, the extraction rates for CS, ReBRS1, ReBRS2, and ReBRS3 samples were 91.25%, 94.02%, 95.81%, and 91.03%, respectively. Correspondingly, the plastic recovery percentages for these samples when using the 4:1 1P and Hp solvent mix were 88.29%, 84.66%, 80.24%, and 84.4%, respectively. Recovery of plastic was consistent across all conditions, and >80% of clean ABS plastics were recovered in each run (Fig. 4(b)). Mass loss occurred due to BFR and additive dissolution and experimental losses during collection. Small particle size leads to better extraction efficiency (Fig. 4(c)). Earlier studies have reported extraction efficiencies <85% [42,48,49]. MAE with a suitable solvent can be a viable option for the recovery of clean plastics. High-capacity microwave ovens are used in the food, paper, timber and textile industries primarily for drying [58], with some design modifications. A high-capacity microwave oven has the potential to serve as a viable technique for the extraction of TBBPA from ABS plastic, particularly in the context of recycling e-waste plastics [59].

Determination of optimized extraction conditions was carried out for

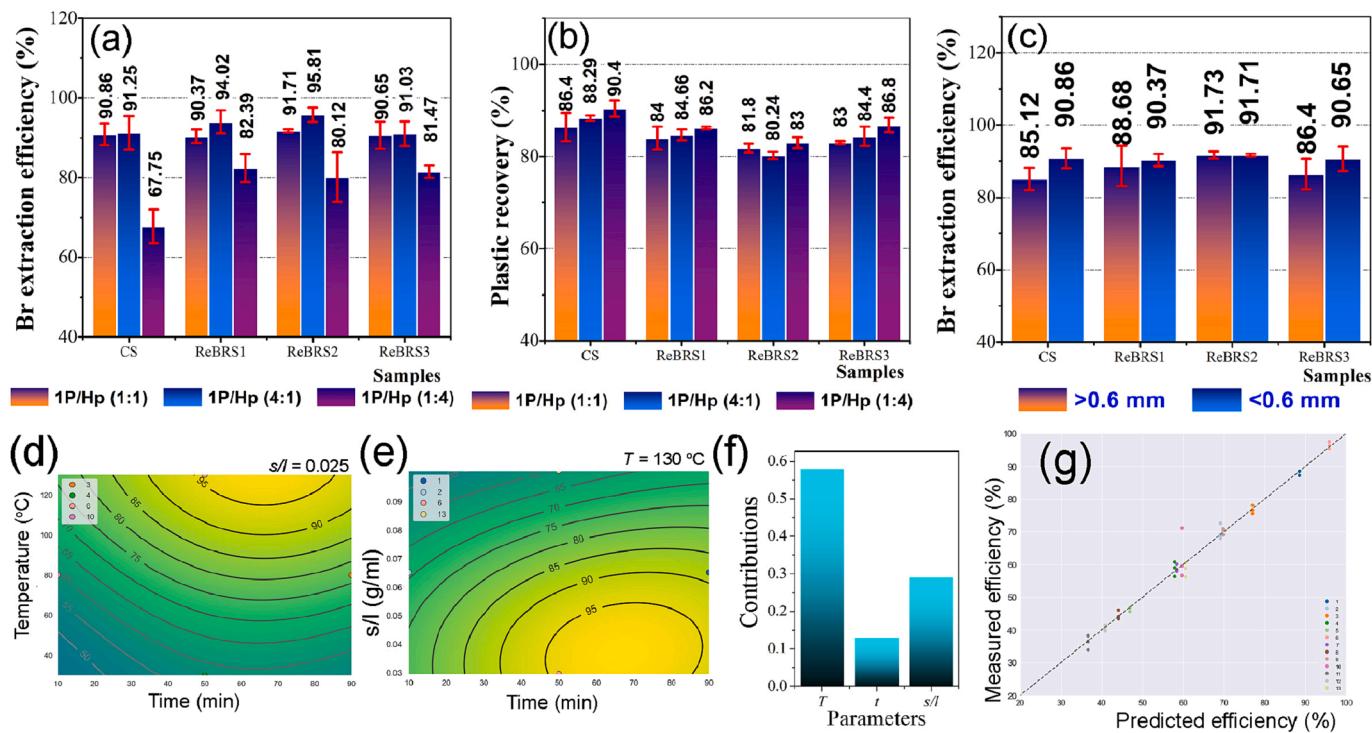


Fig. 4. (a) %Br extraction efficiency, (b) Plastic recovery in the MAE using three solvent mixing ratio of 1P and Hp for particle size < 0.6 mm. (c) Comparison of %Br efficiencies for two particle sizes in MAE with 1P and Hp at 1:1 vol ratio (Experimental condition: $T = 110$ °C, time $t = 90$ mins and $s/l = 0.025$ g/ml). MAE optimization contour plot for (d) T versus t for $s/l = 0.025$ (g/ml); (e) s/l versus t for $T = 130$ °C using SVR to calculate the extrapolated Br extraction efficiencies (%), (f) The analysis of variance (ANOVA) factor evaluation of the optimization, showing the significant contribution of all three parameters, (g) The comparative regression analysis of the predicted output in the response surface and measured efficiency values measured at the DOE.

the solvent mixing ratio of 4:1 (v/v) for 1P to Hp. Experiments were performed according to the DOE, and the Br extraction efficiencies (%) are reported in Table 2S. The response surfaces based on DOE were constructed using SVR with selected hyperparameters. Two contour plots of the predicted output at $s/l = 0.025$ and $T = 130$ °C are shown in Fig. 4(d) and Fig. 4(e), respectively. Complete contour plots are shown in Fig. 6S. The analysis of variance and factor evaluation is shown in Fig. 4(f), indicating that T is the most influential in terms of BFR extraction, compared to the s/l and t . A temperature in the range of 110–130 °C, time in the range of 60 to 70 mins, and s/l ratio of 0.025 give the maximum extraction efficiency. Hence, the predicted output in the response surfaces matches well with experimental observations (Fig. 4(g)).

After MAE, a comparative evaluation of UAE and DR techniques was conducted. A 4:1 (v/v%) mixing ratio of 1P and Hp was used for UAE. Table 3 presents the Br extraction efficiencies (%) and the corresponding plastic recoveries (%) obtained from all three processes experimentally. The results obtained from the UAE technique were found to be like those obtained through MAE, though a minor decrease in plastic recoveries was observed, possibly attributed to high agitation leading to polymer dissolution. In contrast, the DR method resulted in almost complete elimination of BFR, albeit at the expense of a substantial reduction in the amount of recovered uncontaminated plastics (Fig. 5(b)). The solution

may contain a considerable amount of plastic material, particularly low molecular weight ABS, that may remain dissolved [60]. Low extraction efficiency (50–60%) for TBBPA from HIPS was reported by Vilaplana, et al. [51] using UAE. Guo, et al. [61] used d-limonene as solvent and n-propanol as precipitant (anti-solvent) to recover HIPS from e-waste plastic sample with decaBDE as BFR and achieved only ~ 88% BFR removal efficiency in the DR process. Table 4 showcases several previously documented results pertaining to the extraction of bromine (Br)/ BFR from e-waste plastics. However, most of these studies lack comprehensive data regarding plastic recovery. In contrast, the results obtained from the present study demonstrate high Br extraction efficiency over the reported finding and yield a substantial quantity of recyclable ABS plastics.

3.3. Post extraction sample characterisation

The Br content of the plastic samples was the only restricted element present, as indicated in XRF analysis shown in Table 5S. Following extraction, the recovered plastic samples demonstrated a significant reduction in Br levels across all three methods. Furthermore, noteworthy alterations in the concentrations of Ca, Ti, and Zn were observed.

Compiled results of FTIR-ATR, DSC and TGA (DTG) of the untreated plastic samples were added to the supporting information (Fig. 7S). The

Table 3

Optimum Br Extraction efficiency (%) and the plastic recovery (%) from the across all three-process obtained during experiment.

Samples	Br extraction efficiency (%)			Recovered plastics (%)		
	MAE	UAE	DR	MAE	UAE	DR
CS	91.25 ± 4.2	91.82 ± 2.1	97.86 ± 0.78	88.29 ± 0.6	85.4 ± 1.5	83.2 ± 2.31
ReBRS1	94.02 ± 2.89	93.92 ± 0.9	98.15 ± 1.45	84.66 ± 1.2	82.4 ± 0.8	66.45 ± 1.6
ReBRS2	95.81 ± 1.8	93.62 ± 1.5	97.92 ± 0.8	80.24 ± 0.8	84 ± 1.9	69.035 ± 2.1
ReBRS3	91.03 ± 3.1	92.4 ± 2.55	97.56 ± 0.9	84.4 ± 2.1	76 ± 2.7	69.24 ± 3

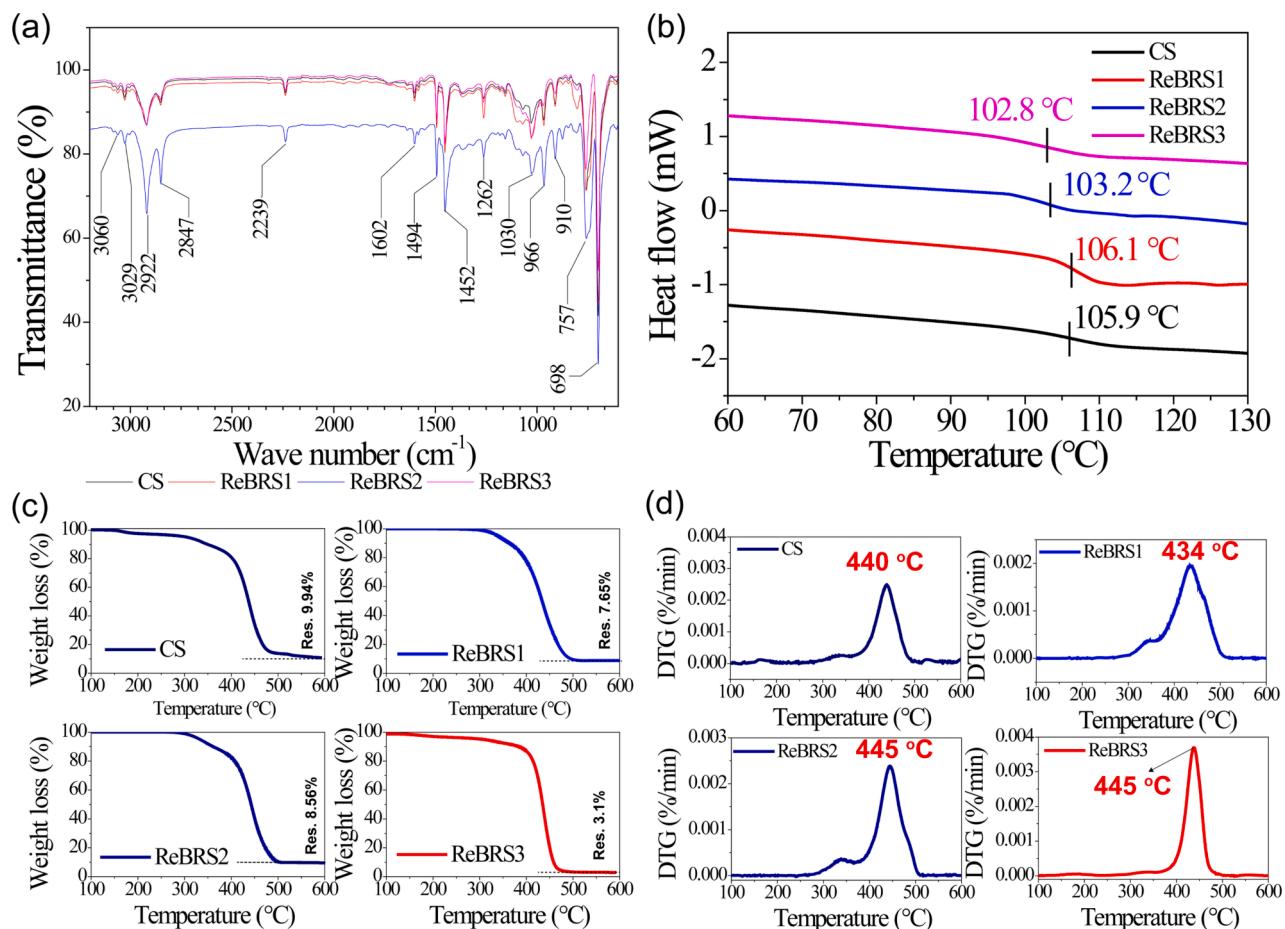


Fig. 5. (a) FTIR spectra with the relevant absorption peaks (absorption band assignment can be found in Table 6S) (b) DSC analysis of each sample showing the T_g , (c) TGA and (d) DTG profiles of the post MAE samples. (Experimental condition: $T = 110$ °C, time $t = 90$ min and $s/l = 0.025$ g/ml with 1P and Hp in 4:1).

Table 4
Previous reported Br extraction from e-waste plastic using various processes and solvents.

Plastic	BFR	Process/solvent	Br extraction efficiency (%)	References
HIPS	TBBPA	Process: MAE	67.64	[42]
HIPS	DecaBDE	$T = 130$ °C, $t = 30$ min, $s/l = 0.05$ g/ml Solvent = Isopropanol/MeOH, 1:1 v/v	15.96	
HIPS	TBBPA	Process: MAE	90.05	
HIPS	DecaBDE	$T = 130$ °C, $t = 60$ min, $s/l = 0.05$ g/ml Solvent = Isopropanol/hexane, 1:1 v/v	21.56	
HIPS	TBBPA	Process: pressurized liquid extraction $T = 150$ °C, (1500 psi) Solvent = Isopropanol/hexane, 1:1 v/v	81.8	[51]
HIPS	TBBPA	Process: pressurized liquid extraction $T = 110$ °C, (1500 psi) Solvent = Isopropanol/MeOH, 1:1 v/v	58.02	
HIPS	DecaBDE	Dissolution and reprecipitation Solvent: d-limonene Precipitant: n-propanol	87.93	[61]

FTIR-ATR spectra of the CS/Real samples are consistent with those of pure ABS (Fig. 3S(b)). On the other hand, values of glass transition temperatures (T_g) of the BFR laden plastics are lower than the T_g values of pure ABS plastics which are around 105–107 °C [62,63]. The T_g of the CS, ReBRS1, ReBRS2 and ReBRS3 are 99.7, 93.7, 92.2 and 91.8 °C, respectively. The two-step degradation profile can be seen in Fig. 7S(d) for the CS/Real samples with a peak degradation temperature at 427 °C, 443 °C, 436 °C, 443 °C for CS, ReBRS1, ReBRS2 and ReBRS3, respectively. For post MAE recovered plastics, the FTIR-ATR, DSC, and TGA (DTG) analysis are shown in Fig. 5. Typical ABS absorption bands are

distinctively present in the FTIR-ATR spectra (Fig. 5(a)) as demonstrated when compared bands from treated samples with those of pure ABS (Fig. 3S(b)). A detailed band assignment for the IR absorption bands associated with ABS/TBBPA is listed in Table 6S. Precisely, the C-H stretches of the aliphatic and aromatic bonds are clearly visible in the range of 3200 – 3000 cm^{-1} and 3000 – 2800 cm^{-1} , respectively [64]. A well-defined peak at 2239 cm^{-1} represents the C≡N stretch of acrylonitrile and the ring structure of styrene appears at absorption bands of 1602 and 1494 cm^{-1} . The other indicators of aromatic ring are seen at 1452 cm^{-1} (-CH₂ scissoring, ring C-H), 757 cm^{-1} and 696 cm^{-1} (ring

bends) [65]. The bond deformation of the C-H bond adjacent to the double bond of the butadiene from ABS is seen at 967 and 910 cm⁻¹. The narrow peak at 1262 cm⁻¹ corresponds to the -OH vibration due to the presence of trace amounts of leftover TBBPA [66]. Furthermore, the T_g values of the post MAE samples improve significantly and approach the T_g values of the commercial ABS (~105 °C) [67]. The thermal degradation characteristics of the plastics recovered after microwave-assisted extraction (MAE) demonstrated a comparable peak degradation temperature to that of the reference ABS plastics, with a temperature proximity of 440 °C observed under non-isothermal heating conditions of 20 °C/min for all samples (Fig. 5(d)) [68]. Therefore, the disappearance of the peak associated with TBBPA in the DTG analysis following MAE did not significantly alter the properties of the polymer. In summary, all the properties of ABS, which was treated with MAE, were consistent with those of the reference polymer.

The characterization results of the recovered polymers after UAE and DR processes are shown in Fig. 8S and Fig. 9S, respectively. The chemical structure of the polymer, as shown by the FTIR-ATR spectra and T_g from DSC, appears to be consistent with pure ABS. No significant changes appeared in the TGA/DTG plot of the samples recovered from UAE.

A slight distortion in the DTG profile can however be seen in recovered samples of the DR process (Fig. 9S(c)). Changes in the peak degradation temperature can be seen, particularly for the ReBRS2, which changes from 436 °C to 465 °C. This could be due to the removal of the stabilizing additives during the dissolution process. The GCMS peaks from the MAE and DR processes are shown in Fig. 10S and Fig. 11S, respectively, along with the likely components discovered by searching the Mass spectral library. TBBPA appeared in all the solutions. In addition to that, traces of phenolic compounds, aromatic compounds (isoquinolines), and organic acids were also present in the solutions, which could be attributed to the various additives, such as dyes, antifungals, antioxidants, and anti-aging compounds mixed within the plastics. The presence of Butylated hydroxytoluene (BHT) in the solvents from the DR process can be attributed to the usage of THF for dissolution, as BHT is a stabilizer commonly found in THF solvents. Among the two solid-liquid extraction processes, MAE appeared to be better than UAE in terms of plastic recovery yields. However, both processes have challenges in terms of scale-up and implementation. UAE requires external heating, while MAE uses microwaves to heat the system. Management of heat and pressure is simpler in MAE compared to UAE because heat can be delivered to the target material via microwave without contact. On the other hand, the DR process can restore the purity of the polymer by reprecipitation from the liquid phase, with reduced polymer yield and abated polymer properties. A DR-like process (CreaSolv®) was developed by Unilever/Fraunhofer Institute for the separation of polymers from a mixed polymer stream of plastics (from different sources) [69,70]. Walker, et al. [71] developed a similar process called solvent-targeted recovery and precipitation (STRAP) process to deconstruct multilayer films and recover the pure resin by performing rigorous solvent screening using HSP and COSMO-RS calculations. Peng, et al. [43] used supercritical CO₂ as an antisolvent for the separation of Deca-BDE (BFR) from HIPS obtained from e-waste. Our results show that MAE can be an excellent method for removing BFR from plastics without affecting plastic properties.

4. Conclusions and perspectives

This study reports the optimal combination of solvents that can be applied to remove the tightly bound BFR additive (TBBPA) from ABS plastics recovered from e-waste [31]. We adopted a rigorous methodology to screen suitable solvent/solvent mixtures that would allow maximum extraction and minimum loss of plastic. The mixture of 1-propanol and heptane was found to be suitable for the extraction of TBBPA from ABS plastics. Non-polar solvents, such as heptane mixed with alcohol, improved the extraction efficiency more significantly than

their individual counterparts. COSMO-RS Calculations showed that both TBBPA and ABS have polar and nonpolar properties on their molecular surfaces that influence the extraction. The targeted solvent-based extraction for recovery of BFR (TBBPA)-free plastics from contaminated e-waste ABS plastics was improved by heating with microwave/ultrasound. In terms of extraction rates, MAE was superior under optimal process conditions as it also preserved/improved the polymer properties of the ABS. The recovery of ABS plastics from the DR process was low compared to MAE /UAE with high extraction efficiency. Such processes could provide a common framework for developing solvent based processes for debromination of BFR laden e-waste plastics. The implementation of a selective and targeted approach is better suited for the recovery of clean plastics and achieving a closed-loop system for recycling e-waste plastics.

Credit authorship contribution statement

Pallab Das: Methodology, Investigation, Formal analysis, Writing – original draft. **Antoine Leybros:** Writing – review & editing. **Jean-Christophe P. Gabriel:** Writing – review & editing. **Chor Yong Tay:** Writing – review & editing. **Jong-Min Lee:** Writing – review & editing, Supervision, Project administration.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cej.2023.144126>.

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