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Comparison between the use of polyether ether ketone and stainless steel columns for ultrasonic-assisted extraction under various ultrasonic conditions

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ABSTRACT

The ultrasound-assisted extraction (UAE) was conducted using the stainless steel (SS) and polyether ether ketone (PEEK) columns and analyzed with high-performance liquid chromatography (HPLC) to understand the mechanism of ultrasound-assisted chromatography (UAC). Empty SS and PEEK columns were used to extract dyes from a fabric under identical conditions with several parameters including the initial ultrasonic bath temperatures (30 °C and 40 °C), ultrasound power intensities (0, 20, 40, 60, 80, and 100 %), ultrasound operation modes (normal and sweep), and ultrasound frequencies (25 kHz, 40 kHz, and 132 kHz) to compare their extraction capabilities. After 30 min of extraction, the amount of extract was determined by HPLC. The PEEK material was significantly affected by ultrasonic radiation compared to the SS material, especially at a higher temperature (40 °C), power intensity (100 %), and frequency (132 kHz) with sweep mode. At a maximum power density of 45 W/L, the extraction effectiveness ratio of PEEK to SS was in the range of 1.8 - 3.9 depending on the specific frequency, initial temperature, and with or without temperature control. The most optimal ultrasound frequencies, in terms of enhancing extraction effectiveness, are in the order of 132 kHz, 40 kHz, and 25 kHz. Unlike the SS material, the PEEK material was more affected by temperature and acoustic effects under identical conditions, especially at 132 kHz ultrasound frequency. In contrast, at lower frequencies of 40 kHz and 25 kHz, no significant differences in the acoustic effects were observed between the PEEK and SS materials. The findings of this study contribute to elucidating the roles of column materials in UAE and UAC.

1. Introduction

Ultrasound is the sound waves with frequencies beyond the human audible range (>20 kHz) and categorized into three frequency ranges: low frequencies (20 kHz – 100 kHz), high frequencies (100 kHz – 1 MHz), and diagnostic ultrasound (1 MHz – 500 MHz) [1]. Among many novel techniques, the ultrasound emerged with its advantages as a rapid, low-cost, non-thermal, environmentally friendly, and easy-to-operate method [2,3]. Therefore, it has attracted considerable research interest and has been widely applied in various fields including food processing, cleaning, sonochemistry, plastic welding, and medical applications [3,4] in laboratory and industrial settings.

Currently, ultrasound-assisted extraction (UAE) is widely used to obtain natural components from plants such as ginseng saponins from ginseng roots [5], phenolic compounds from wheat bran [6], essential oil from garlic [7], polyphenols from black chokeberry [8], oil from flaxseed [9], and lycopene from tomato [10]. The key factors that contribute to the extraction yield in ultrasonic bath systems include but not limited to power, extraction time, ultrasound frequency, bath water temperature, solvent (composition and pH), sample position (vertical or horizontal) in the vessel, type of extraction vessel, etc. [5–10].

The ultrasound radiation is provided by either ultrasonic bath or ultrasonic probe in most laboratory environments. An ultrasonic probe is approximately 100-fold more powerful than the ultrasonic bath [11] because its ultrasound intensity is focused on to a smaller area by direct injection of the probe tip into the extraction chamber [12–14]. In contrast, the ultrasound intensity of an ultrasonic bath is easily attenuated by the bath liquid, and the cavitation is often not achieved in the

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submerged extraction vessel [13,15]. Nevertheless, the ultrasonic bath is more favorable, economical, and easier to handle.

Typically, the ultrasonic bath system is utilized with a flask or beaker made of plastic, glass, or stainless steel (SS) as an extraction vessel, which are prone to attenuation of ultrasonic energy. However, extractability can be enhanced by treating the sample in the SS vessel with a low frequency ultrasound as an intermediate combination step [16]. Due to the attenuation of energy involved in the ultrasonic bath system, selection of vessel material requires careful multivariate consideration. Nevertheless, to the best of our knowledge, previous studies have not investigated the effects of different vessel materials on UAE.

The ultrasonic bath system requires a use of an extraction vessel to propagate the ultrasonic radiation. Several studies have optimized the extraction yield with respect to varying ultrasound frequencies using a specific type of vessel [17–20]. Previous UAE studies investigated the effects of 25 kHz and 40 kHz ultrasound frequencies on the extraction of glycyrrhizic acid from licorice [17], piperine from *Piper longum* [18], and ursolic acid from *Ocimum sanctum* [19]. These extractions were carried out in the glass vessel, and the results cohesively demonstrated greater extraction effectiveness with the ultrasound frequency of 40 kHz than 25 kHz. In the study by Dong et al. [20], a conical beaker was used as a vessel to load *Salvia miltiorrhiza* roots for extraction of salvianolic acid B under three different ultrasound frequencies: 28 kHz, 45 kHz, and 100 kHz. The optimal ultrasound frequency for the extraction was 45 kHz. Likewise, Ma et al. [21] used three ultrasound frequencies of 20 kHz, 60 kHz, and 100 kHz to extract hesperidin from Penggan (*Citrus reticulata*) peel placed in a glass beaker, in which the optimum frequency was 60 kHz. Above prior studies have consistently demonstrated the significance of optimal ultrasound frequency to maximize extraction effectiveness in UAE.

In the field of chromatography, ultrasound [22–27] and other external fields such as magnetic, optical, electric, and temperature [28–40] are used to manipulate chromatographic retention to optimize chromatographic separation and selectivity. For example, Cheng et al. [22] applied 20 kHz ultrasound frequency to agitate strong acidic and basic resins and observed an increase in ion-exchange rate. Okada [23] found that 47 kHz ultrasound frequency either reduced or enhanced the retention of large, less solvated ions and small, more solvated ions, respectively. This further supports the potential use of ultrasound in controlling the ion-exchange chromatographic retentions for simple ions. Oszwaldowski and Okada [24] tested polytetrafluoroethylene (PTFE) separation column in the ultrasonic bath at 35 kHz frequency and concluded ultrasound as an effective external factor which can control chromatographic retention and ionic interactions. Furthermore, our previous studies have introduced the idea of employing 25 kHz and 42 kHz ultrasound frequencies onto the SS column for chiral separation and demonstrated that ultrasound coupled with varying temperatures improved enantioselectivity and chiral separation efficacy while reducing the analysis time [25–27].

The SS column is widely used for the high-performance liquid chromatography (HPLC) and polyether ether ketone (PEEK) column is generally used for the ion-exchange chromatography. Based on the previous studies, investigating the effect of the ultrasound on UAE using various frequencies and column materials is subject to great challenges. This study attempts to define the roles of the column material and ultrasound frequency in ultrasound-assisted chromatography (UAC) using the SS and PEEK columns by investigating the mechanism of ultrasound effect in the UAE. The effect of the ultrasound on the UAE of the navy dye was examined by comparing the UAE efficiency of the SS and PEEK columns under varying ultrasound frequency, initial temperature, power intensity, and operation mode. Ultrasound was provided by the three ultrasonic bath systems of different frequencies (25 kHz, 40 kHz, and 132 kHz) with multiple controlled parameters: initial temperatures (30 °C and 40 °C), power intensities (0, 20, 40, 60, 80, and 100 %), and operation modes (normal and sweep).

2. Materials and methods

2.1. Reagents and instruments

Navy dyed polyethylene terephthalate (PET) and navy standard dye (disperse Navy MPL; commercial name) were purchased from Sunshine Color Tech Co., ltd (Shanghai, China). Extraction solvent acetone (99.5 %) was purchased from Duksan Pure Chemical Co., ltd. (Ansan, South Korea) and HPLC grade solvents 2-propanol and *n*-hexane were purchased from J. T. Baker (Center Valley, PA, USA). Two empty HPLC columns (250 mm × 4.6 mm): an SS column (Phenomenex, Torrance, CA, USA) and a PEEK column (Thermo Fisher Scientific, Waltham, MA, USA). A 0.45 μm nylon syringe filter from Hyundai Micro Co., ltd (Seoul, South Korea) was used. Three ultrasonicators with different ultrasound frequencies were used: (1) the S8525-12 ultrasonicator (500 W, 25 kHz) and CH1012-25–12 ultrasonic tank with internal dimensions of 254 mm × 304 mm × 254 mm (Branson Ultrasonics Corp., Danbury, CT, USA); (2) the SD-D400H ultrasonic bath (400 W, 40 kHz) with internal dimensions of 600 mm × 350 mm × 315 mm (SD-Ultrasonic Co. ltd., Korea); (3) the MW 500 HMI Crest ultrasonic cleaner (500 W, 132 kHz) with internal dimension 355 mm × 257 mm × 240 mm (Crest Ultrasonics Corp., USA). All ultrasonicators were equipped with intensity and temperature controllers. The temperature inside the ultrasonic bath was measured with a digital thermometer (SDT142S, Summit Co., ltd., Incheon, South Korea). The HPLC system (Waters 2690 Separations Module) consisted of a Waters 996 photodiode array detector and an autosampler (Water Corporation, Milford, MA, USA).

2.2. HPLC analysis

2.2.1. Standard dye analysis

The navy standard dye powder was dissolved in acetone to prepare the 1000 mg/L stock solution, and then diluted 2, 10, and 20 times to prepare four standard dye samples. All standard samples were filtered through the nylon syringe filter before HPLC analysis. The four navy standard dye samples (50 mg/L, 100 mg/L, 500 mg/L, and 1000 mg/L) were analyzed via HPLC to formulate the calibration curve. The quantitative analysis was calculated using the linear regression equation after examining the Pearson correlation coefficient. Silica column (YMC-Pack SIL/S-5 μm/12 nm, 4.6 mm ID × 250 mm length, YMC Co., ltd., Kyoto, Japan) was used as a stationary phase. The mobile phase solvent, composed of 2-propanol and *n*-hexane in 1:9 ratio, was used at a flow rate of 1.0 mL/min. The column and sample temperatures were set to 25 °C and the sample injection volume was 10 μL with detection at 600 nm wavelength.

2.2.2. Fabric sample analysis

The fabric (200 ± 0.5 mg) was cut into long, thin strips and inserted into the empty SS and PEEK HPLC columns. Subsequently, 2.00 mL of acetone was added to each column before closing the columns. After 30 min of UAE (see Section 2.3) in the ultrasonic bath, all liquids and fabrics inside each column were transferred into separate 20 mL vials. The extractants including the fabric strips were stirred carefully before sampling 0.700 mL and diluting it with 0.700 mL acetone in a 5 mL vial. The samples were filtered through the nylon syringe filter prior to HPLC analysis. Each column was thoroughly cleaned with acetone before and after each experiment to avoid contamination, which could affect the results. The HPLC conditions were identical to those of the standard dye analysis (Section 2.2.1).

2.3. Ultrasound-assisted extraction

2.3.1. 25 kHz UAE effectiveness comparison between PEEK and SS columns

Each column (SS and PEEK) with fabric strips and acetone solvent was placed in the tube rack and then immersed into the ultrasonic water

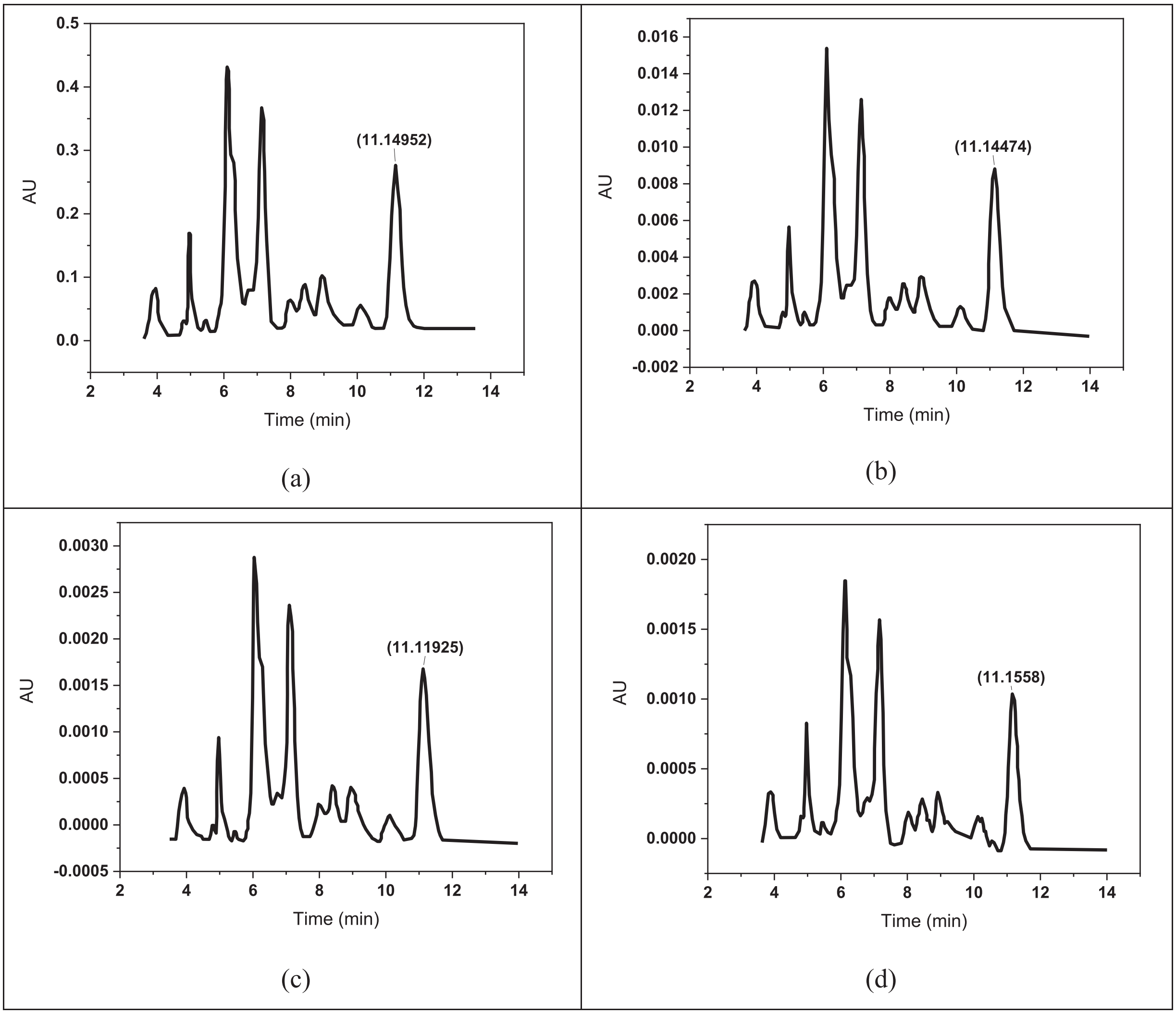


Fig. 1. Chromatograms of navy standard dye samples in HPLC analysis via silica column. Column: YMC-Pack SIL/S. Mobile phase: 2-propanol in *n*-hexane (1:9) with 1.0 mL/min flow rate, 10 μL sample injection volume, detection wavelength at 600 nm, and column temperature at 25 °C. (a) 1000 mg/L, (b) 500 mg/L, (c) 100 mg/L, and (d) 50 mg/L.

bath. The position of the columns in the tube rack and their distance from the bottom of the ultrasonic bath system were kept constant throughout this study. The extractions were conducted for 30 min at an ultrasound frequency of 25 kHz in the ultrasonic bath at different initial temperatures (30 °C and 40 °C). The initial temperature of the ultrasonic bath was maintained with a deviation of ± 1 °C by adding or draining cold or hot water during the extraction. The water volume (11 L) of the ultrasonic bath was kept constant. The output power intensity was set to 0 % (non-sonication), 20 %, 40 %, 60 %, 80 %, and 100 % of 500 W with respect to each experimental condition.

2.3.2. *Effect of temperature on PEEK and SS columns using 25 kHz ultrasound frequency*

Here, identical procedure discussed in Section 2.3.1 was integrated for the 25 kHz ultrasonic bath system. However, the ultrasonic bath temperatures of 30 °C and 40 °C were not controlled to remain constant once the ultrasound application (from 20 % of 500 W) initiated. For each experiment, the output power was fixed to 0 %, 20 %, 40 %, 60 %, 80 %, or 100 % of 500 W, respectively. After completion of the extraction process, the water temperature increment for each experiment was recorded using a digital thermometer.

2.3.3. *Effects of different ultrasound frequencies on PEEK and SS columns*

Ultrasound frequencies of 40 kHz and 132 kHz were tested in addition to 25 kHz. To accommodate for power differences between the ultrasonic bath systems (400 W and 500 W), the acoustic power density was calculated to determine the standardized power-to-water volume ratio. In this study, the acoustic power density was set to 45 W/L; the corresponding ratios were 500 W/11 L and 400 W/9 L. The procedure was identical to Section 2.3.1, except that output power intensities of 0 %, 40 %, 60 %, 80 %, and 100 % were used.

2.3.4. *Effect of temperature on PEEK and SS columns at different ultrasound frequencies*

Section 2.3.2 procedure was used with corresponding water volumes for each ultrasonic bath system previously determined in Section 2.3.3. However, the output power intensities were adjusted to 0 %, 40 %, 60 %, 80 %, and 100 % for each test. Lastly, the final water temperature at the end of each extraction was documented.

2.3.5. *Comparison of extraction effectiveness between different operation modes at 25 kHz ultrasound frequency*

The ultrasonic bath system (25 kHz) has two operation modes: normal and sweep. The operation mode was set prior to each experiment. Once the ultrasonic bath reached the predetermined initial

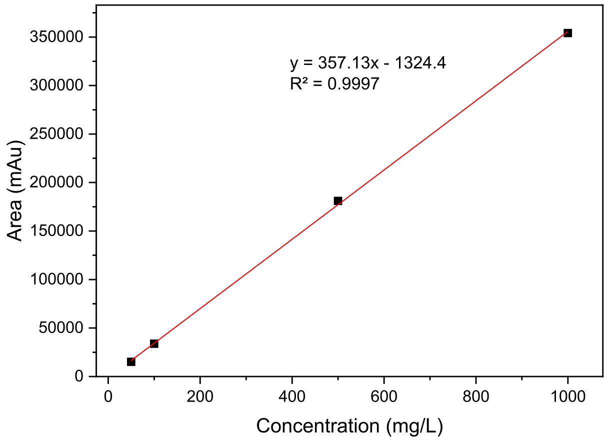


Fig. 2. The calibration curve of the amount of navy dye.

Table 1
Extraction amounts of navy dye at 30 °C and 40 °C with temperature control at 25 kHz ultrasound frequency[a].

Ultrasound power intensity (%)	30 °C		40 °C	
	PEEK	SS	PEEK	SS
0	365.5	309.8	949.8	832.7
20	442.7	363.3	1281.1	927.8
40	494.1	372.4	1818.1	939.4
60	603.5	381.1	2011.0	946.5
80	687.4	387.9	2098.2	951.9
100	713.5	393.1	2169.2	963.7

[a] Unit of the amount of Navy dye is mg/L.

temperature, the tube rack with SS and PEEK columns was immersed into the bath. All extraction experiments were carried out under the same conditions as those discussed in Sections 2.3.1 and 2.3.2. Upon completion of the extraction for each mode, the final water temperature for each experiment was recorded using a digital thermometer. All experiments in this study were repeated three times.

3. Results and discussion

3.1. Calculation of the amount of extracted navy dye

To calculate the amount of navy dye extracted from the fabric, standard navy dye samples of 50 mg/L, 100 mg/L, 500 mg/L, and 1000 mg/L were analyzed with HPLC as shown in Fig. 1.

Navy dye peaks detected at a wavelength of 600 nm appeared at 11.15 min. As shown in Fig. 1, as the concentration decreased from 1000 mg/L to 500, 100, and 50 mg/L, the peak area of the navy dye at 11.15 min was reduced. Therefore, the standard calibration curve was generated using the areas of the peaks to quantify the amount of extracted navy dye using the HPLC analysis. A linear regression equation was obtained from the plot of the standard dye concentration (mg/L) against the peak area. Linearity of the calibration curve is illustrated by the coefficient R^2 in Fig. 2.

From Fig. 2, the concentrations of the navy dye were calculated via $y = 357.13x - 1324.4$ with $R^2 = 0.9997$, where y is the peak area of the navy standard dye and x is the navy standard dye concentration. The $R^2 = 0.9997$ demonstrates high linearity, thus the analysis method accurately quantifies the amount of extracted navy dye. This equation was used in the quantitative analysis of the amount of navy dye extracted from the fabric.

3.2. Comparison of UAE effectiveness between the PEEK and SS columns at 25 kHz ultrasound frequency

The ultrasound frequency of 25 kHz was employed to compare the UAE effectiveness between the PEEK and SS columns. The ultrasonic bath temperature was controlled to maintain its initial temperature (30 °C or 40 °C) for each output power setting. The UAE results at 25 kHz frequency are shown in Table 1.

Under non-sonication, the extracted amounts of navy dye via PEEK column were 365.5 and 949.8 mg/L at 30 °C and 40 °C, respectively, whereas the extracted amounts via SS column were 309.8 and 832.7 mg/L at 30 °C and 40 °C, respectively. The amount of extracted navy dye between the PEEK and SS columns changed as ultrasound was applied during the extraction. With increasing ultrasound intensity, the extraction amount of navy dye for the SS column slightly increased and significantly increased for the PEEK column. At the maximum ultrasound intensity (100 %), the extracted amounts of navy dye for PEEK column were 1.8 and 2.3 times higher than those of the SS column at 30 °C and 40 °C initial temperatures, respectively. This demonstrates that the SS material is less affected by the ultrasound effect compared to the PEEK material. The PEEK column's better extraction performance, in contrast to the SS column, under controlled temperature conditions can be attributed to its physical properties such as a microstructure, hardness, elasticity, and plasticity yielding different acoustic resistances. At 20 °C, the acoustic resistances of water, steel, and most plastics are 1.48×10^6 Pa·s·m^{-1}, 4.54×10^7 Pa·s·m^{-1}, and $2 \sim 3 \times 10^6$ Pa·s·m^{-1}, respectively [24]. Accordingly, the acoustic resistance of the SS material is 30 times higher than the water and 15 ~ 23 times higher than the PEEK material. Thus, the ultrasound is transmitted more easily from the water to the PEEK material than to the SS material. Therefore, when the ultrasound intensity increases, the extraction efficiency is higher with the PEEK material.

Okada [23] and Oszwaldowski and Okada [24] used polytetrafluoroethylene (PTFE) columns, which is a similar type of plastic to the PEEK material used in this study. Their studies utilized a low ultrasound output power (60 W or 200 W) at 25 °C to distinguish the effects of ultrasound from those of the temperature. The results showed that the ultrasonic effect can modify the separation selectivity in ion-exchange chromatography. The ultrasound radiation, rather than temperature, has a dominant effect on reducing or enhancing the retention time depending on the larger, less-solvated ions or smaller, more-solvated

Table 2
Extraction amounts of navy dye at 30 °C and 40 °C without temperature control at 25 kHz ultrasound frequency[a].

Ultrasound power intensity (%)	30 °C				40 °C			
	PEEK	SS	ΔT (°C)	P_a (W)	PEEK	SS	ΔT (°C)	P_a (W)
0	365.5	309.7	0	0	949.8	832.7	0	0
20	498.9	368.5	+0.8	20.5	1368.7	976.0	0	0
40	832.6	377.9	+4.0	102.7	2268.6	1026.4	+2.0	51.3
60	1137.9	413.8	+6.0	154.0	2691.7	1254.8	+4.2	107.8
80	1421.6	539.8	+8.7	223.3	3162.3	1419.9	+7.0	179.7
100	1902.7	785.6	+11.4	292.6	3460.4	1693.1	+9.7	249.0

[a] Unit of the amount of Navy dye is mg/L. ΔT represents the change in bath temperature after the 30 min of extraction. P_a is the actual acoustic power in the ultrasonic bath.

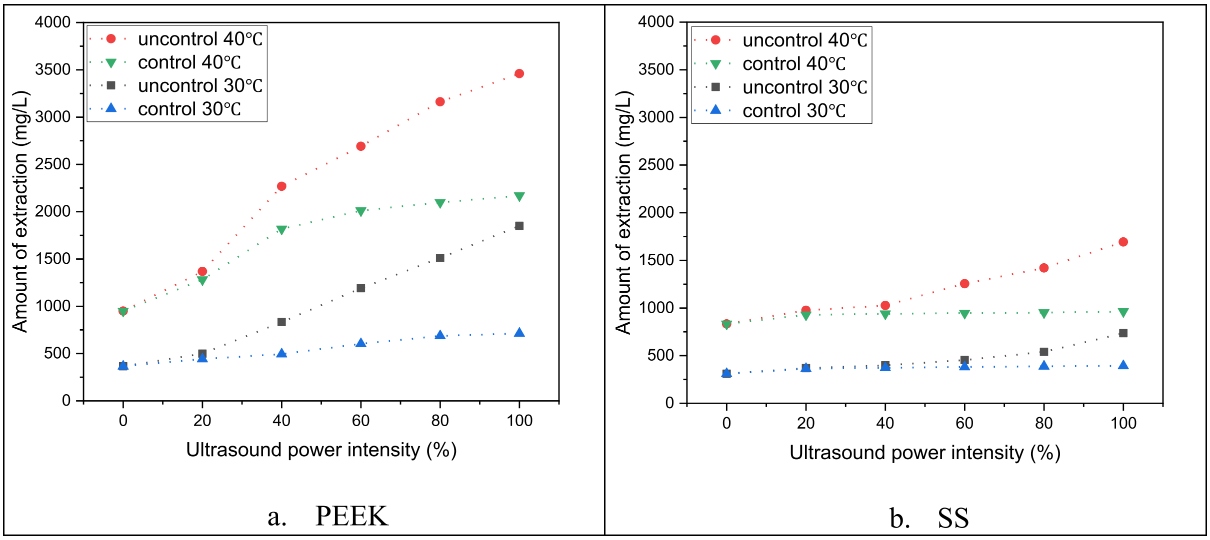


Fig. 3. Comparison of extraction amount of navy dye at 30 °C and 40 °C with and without temperature control using (a) PEEK and (b) SS columns.

ions, respectively [23]. However, in our previous studies on ultrasound-assisted chiral chromatography [25–27], the SS column under the ultrasound output power of 135 W at different temperatures (10, 20, 25, 30, 40, 45, 50, and 60 °C) demonstrated small differences in extraction efficiency between the sonic and non-sonic conditions. Under ultrasound radiation, the elution time decreased at all temperatures and the enantioselectivity was improved at higher temperatures (45, 50, and 60 °C) [25,26]. Previous study on ultrasound-assisted chiral separation utilized the ultrasound reduction technique at a high temperature, in which the elution of the first peak was accelerated with the ultrasound, and then the elution of the second peak was unaltered by removing the ultrasound [27]. The separation and resolution of the chiral compound slightly improved as the distance between the two enantiomer peaks increased. Overall, both PEEK and SS columns demonstrated improved UAE efficiency when the temperature was controlled, however, the UAE appeared to be most effective with the PEEK column.

3.3. *Effect of temperature on the PEEK and SS columns at 25 kHz ultrasound frequency*

To investigate the influence of the temperature on the PEEK and SS column materials, the extraction was carried out without controlling the ultrasonic water bath temperature (30 °C or 40 °C). The results are shown in Table 2.

As the ultrasound power intensity increased, the corresponding extraction efficiencies for both PEEK and SS columns increased. The amounts of extraction substantially increased with ultrasound application from 365.5 mg/L to 1902.7 mg/L (0 to 100 % power intensity) at 30 °C and from 949.8 mg/L to 3460.4 mg/L (0 to 100 % power intensity) at 40 °C for the PEEK column, which corresponds to 5.2- and 3.6-folds increase, respectively. For the SS column, the extraction amounts increased from 309.7 mg/L to 785.6 mg/L (0 to 100 % power intensity) at 30 °C and 832.7 mg/L to 1693.1 mg/L (0 to 100 % power intensity) at 40 °C, which is 2.5- and 2-folds increase, respectively. The ultrasound power intensity can be represented in terms of the acoustics power using the equation (1).

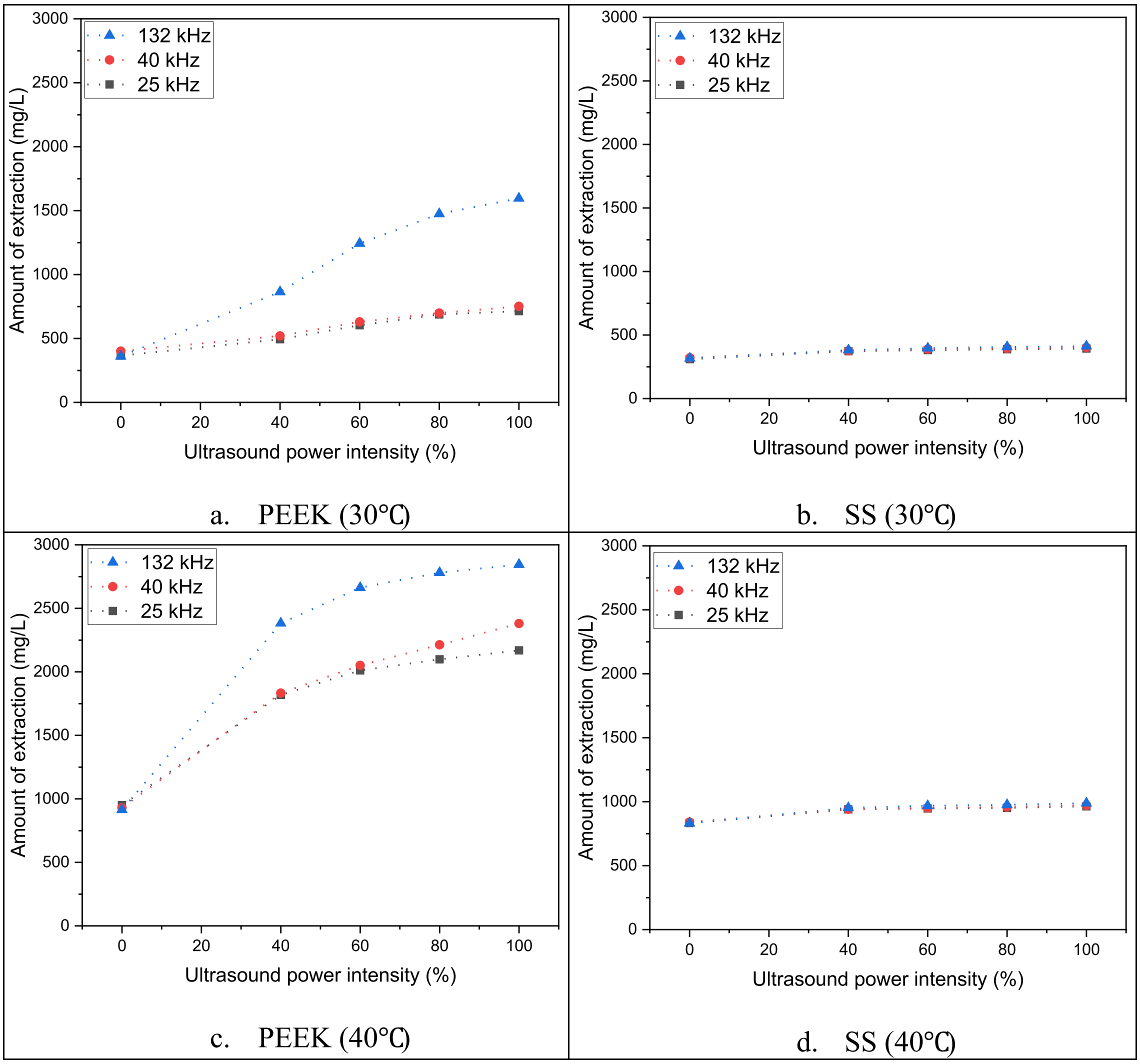
$$P_a = mC\frac{dT}{dt} \tag{1}$$

The actual power delivered to the system is the acoustic power absorbed by the medium, which led to increase in temperature [41,42]. The P_a is actual acoustic power (W) in the medium, m is the mass of the water (kg), C is the specific heat capacity of water (4200 J/kg·K), and dT/dt is the rate of temperature change (K) over a period of time, t (s).

The addition of temperature as an experimental variable noticeably enhanced the UAE efficiency of the SS column. Based on the minimal ultrasound effect on the SS column observed in prior studies [25–27], the enhanced extraction effectiveness of the SS column can be explained by the increased interaction between the sample and the stationary phase resulting from the conversion of ultrasound energy to heat. Furthermore, greater temperature increase was observed with higher ultrasound intensities for both initial temperatures (30 °C and 40 °C). However, at the higher initial temperature (40 °C), the temperature increment was smaller relative to that at the lower initial temperature (30 °C). The temperature increments for 30 °C and 40 °C initial temperatures were not identical with respect to each ultrasound intensity tested. When the ultrasound intensity increased from 0 % to 20 % or more, the average temperature increment was 2.1 °C for 30 °C and 1.9 °C for 40 °C initial temperature conditions. The relationship between the temperature increment and ultrasound intensity can be elucidated by the cavitation phenomenon. Cavitation occurs when the acoustic pressure reaches the cavitation threshold, in which the ultrasound stimulates the formation of microbubbles leading to rapid compressions and expansions that increases the local temperature and pressure within [43]. In addition, the ultrasound intensity is directly proportional to the temperature increment and extraction efficiency, which can be attributed to waves of higher ultrasound intensity creating more bubbles that would collapse [44] and generate shockwaves that disrupt the fabric walls. Therefore, facilitating the penetration of the solvent into the fabric and releasing the dyes from the fabric at a much faster rate [45].

Generally, the positive effects of a higher temperature on viscosity, diffusivity, solubility, and surface tension [46] induce faster thermal motion of the molecules and accelerated solubility and diffusivity of the dyes from the fabric into the solvent. Furthermore, the temperature increment during the extraction could break bonds and affect the fabric structure, and thus facilitating the extraction process [47]. The cavitation intensity decreased when the temperature increased [48], which had a negative effect on the extraction. In addition, water has a maximum cavitation intensity at 35 °C [49]. This is one of the factors responsible for the relatively lower temperature increments at 40 °C initial temperature compared to that at 30 °C initial temperature. However, from a thermal perspective, the higher temperature had a positive effect on extraction efficiency. Conclusively, there is an optimal temperature at which the cavitation and thermal effects synergistically yield positive effects on the extraction process.

The extraction effectiveness of the PEEK and SS column materials based on the amount of extracted dye under different temperature



a. PEEK (30°C)

b. SS (30°C)

c. PEEK (40°C)

d. SS (40°C)

Fig. 4. Effects of different ultrasound frequencies on (a) PEEK at 30 °C, (b) SS at 30 °C, (c) PEEK at 40 °C, and (d) SS at 40 °C.

conditions are organized in Fig. 3.

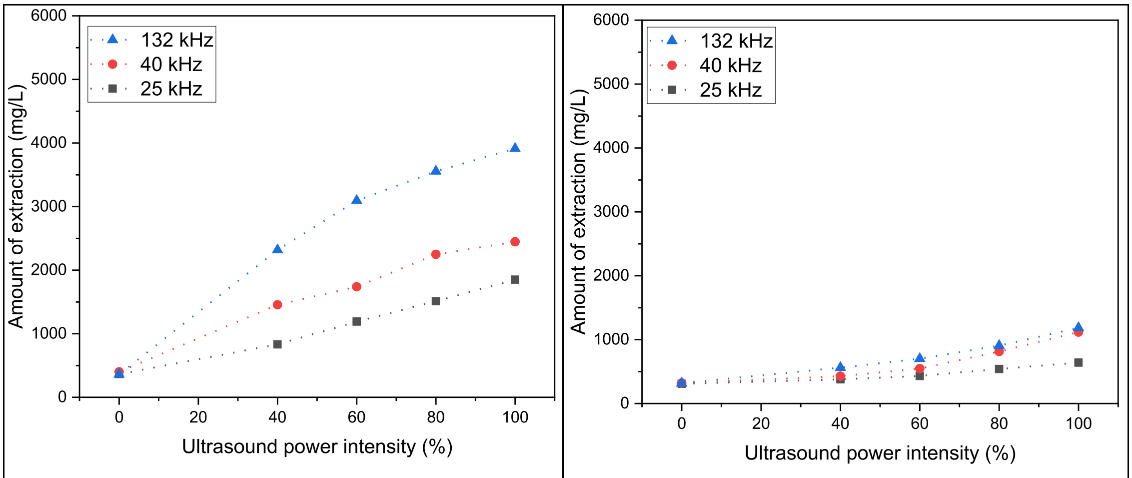
From Fig. 3, the difference in the extraction amounts between the two methods (temperature controlled and uncontrolled) can be clearly observed under identical conditions, regardless of the initial temperatures. Both the PEEK and SS columns demonstrate continuous positive trend of extraction amounts in accordance with increasing power intensity when the temperature is not controlled. The combined effects of the ultrasound and temperature yield the greatest enhancement of extraction efficiency for both PEEK and SS materials. When the temperature is controlled, only the PEEK column shows notable increase in the extraction amounts. In contrast to the PEEK material, the SS material exhibited negligible enhancement in the extraction efficiency with the temperatures controlled. Although the extraction efficiencies of both column materials were affected by the temperature, the effect was more significant for the PEEK material than the SS material under identical conditions.

3.4. Effects of different ultrasound frequencies on PEEK and SS columns

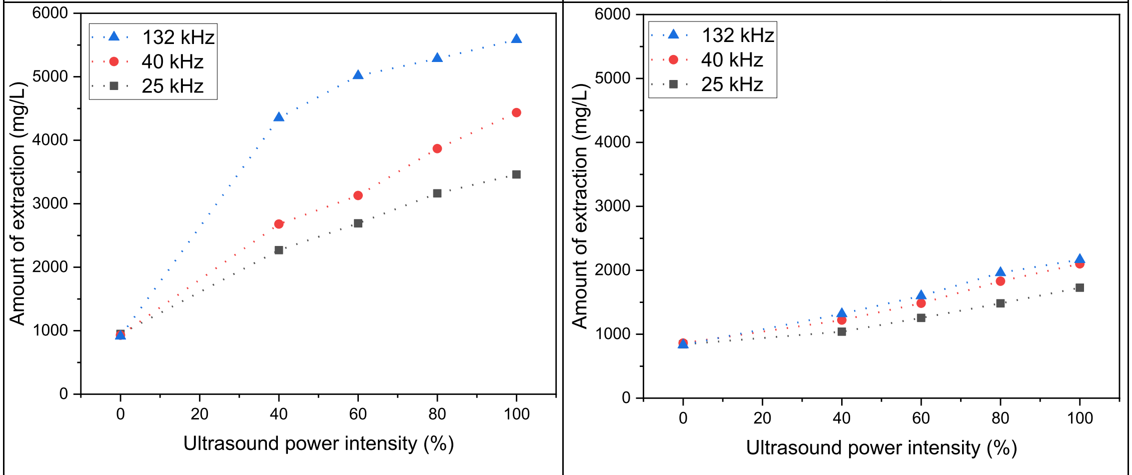
To extensively compare the effects of different ultrasound frequencies on the extraction efficiencies of the PEEK and SS materials, 40 kHz and 132 kHz ultrasound frequencies were examined in addition to 25 kHz with initial temperatures of 30 °C and 40 °C maintained throughout the extraction. The results are shown in Fig. 4.

Regardless of the initial temperatures, the concentration of the extracted dye for the PEEK material was the highest from 40 to 100 % intensities at the highest ultrasound frequency (132 kHz) compared to that of lower ultrasound frequencies (25 kHz and 40 kHz). The extracted dye concentrations demonstrated relatively similar positive trend between the 25 kHz and 40 kHz ultrasound frequencies for the PEEK material at 30 °C and 40 °C. On the contrary, the concentrations of the extracted dye for the SS material were relatively similar among the three ultrasound frequencies at 30 °C and 40 °C, regardless of the ultrasound intensities.

The main underlying mechanism of the results shown in Fig. 4 appears to be a mechanical effect [3]. The mechanical effect of ultrasonic waves occurred after the waves have propagated into the water, resulting in the vibration of the particles in the medium. These mechanical waves may agitate the medium to create a stirring effect, enhancing the movement of the water particles and accelerating the mass transfer as a result. Furthermore, a low ultrasound frequency generates a strong shear or physical force, whereas a high ultrasound frequency generates a weak force [50]. However, a higher ultrasound frequency induces formation of more cavitation bubbles. In addition, larger bubbles are created at lower frequencies with a more violent collapse, and the collapse rate over unit of time is increased at higher frequencies [51]. Therefore, it can be assumed that the high ultrasound frequency of 132 kHz was optimal to increase the cavitation bubbles and collapse rate to promote the enhanced dye extraction under temperature-controlled conditions.



a. PEEK (30℃) b. SS (30℃)



c. PEEK (40℃) d. SS (40℃)

Fig. 5. Temperature effect on PEEK and SS columns using three different frequencies under different temperature conditions for UAE. (a) PEEK at 30 °C, (b) SS at 30 °C, (c) PEEK at 40 °C, and (d) SS at 40 °C.

Table 3
The final temperature increments of the ultrasonic bath water after the extraction[a].

Ultrasound power intensity (%)	30 °C			40 °C		
	25 kHz	40 kHz	132 kHz	25 kHz	40 kHz	132 kHz
40	+4.0	+9.0	+6.5	+2.0	+5.6	+4.6
60	+6.0	+11.0	+9.7	+4.2	+9.1	+7.4
80	+8.7	+14.1	+12.0	+7.0	+12.6	+9.8
100	+11.4	+18.0	+16.5	+9.7	+13.8	+12.0

[a] Extraction time: 30 min.

Prior studies have utilized various ultrasound frequencies including 35 kHz, 42 kHz, and 47 kHz to manipulate UAC [23–26]. In this study, varying the ultrasound frequencies did not hinder the corresponding acoustic effects. Rather, the types of column material and bath temperatures dictated the extraction effectiveness.

3.5. *Effect of temperature on the PEEK and SS columns at different ultrasound frequencies*

The effect of temperature on the extraction effectiveness of the PEEK and SS columns at three ultrasound frequencies without the initial temperatures controlled is illustrated on Fig. 5.

The extraction efficiency of the PEEK column was significantly increased in response to the increasing ultrasound frequencies and intensities compared to that of the SS column. Based on the Fig. 5, the descending order of extraction effectiveness for both PEEK and SS columns is 132 kHz, 40 kHz, and 25 kHz. Previous studies have reported a positive correlation between the extraction efficiency and ultrasound frequency [17–21], which corresponds to the observations of this study.

To investigate the effect of ultrasound frequencies on the ultrasonic bath water temperature, the final water temperature after 30 min of extraction without temperature control was recorded for each frequency at the specified intensities in Table 3.

With increasing ultrasound intensity, higher temperature increment was observed at all ultrasound frequencies. Although Fig. 5 illustrates highest extraction effectiveness achieved at 132 kHz ultrasound frequency, the highest temperature increment was observed at 40 kHz. It is believed that the mechanical effects and cavitation phenomenon are the main contributing factors of the observations. However, a significant cavitation effect cannot be achieved by the bubbles alone. Prior studies have proven that if the ultrasound frequency is matched with the natural resonance frequency of the bubble, the ultrasonic energy would reach its highest [52–58]. The presumption was that the radius of the bubble had changed. Huang et al. formulated and expressed the relationship

Table 4
The ratio of extraction amount of PEEK and SS columns at ultrasound power intensity of 100%[a].

Temperature method	30 °C			40 °C		
	25 kHz	40 kHz	132 kHz	25 kHz	40 kHz	132 kHz
Control	1.8	1.9	3.9	2.3	2.5	2.9
Uncontrol	2.9	2.2	3.3	2.0	2.1	2.6

[a] Data was calculated by dividing the extractant amount in PEEK by that in SS material for UAE, its unit: times.

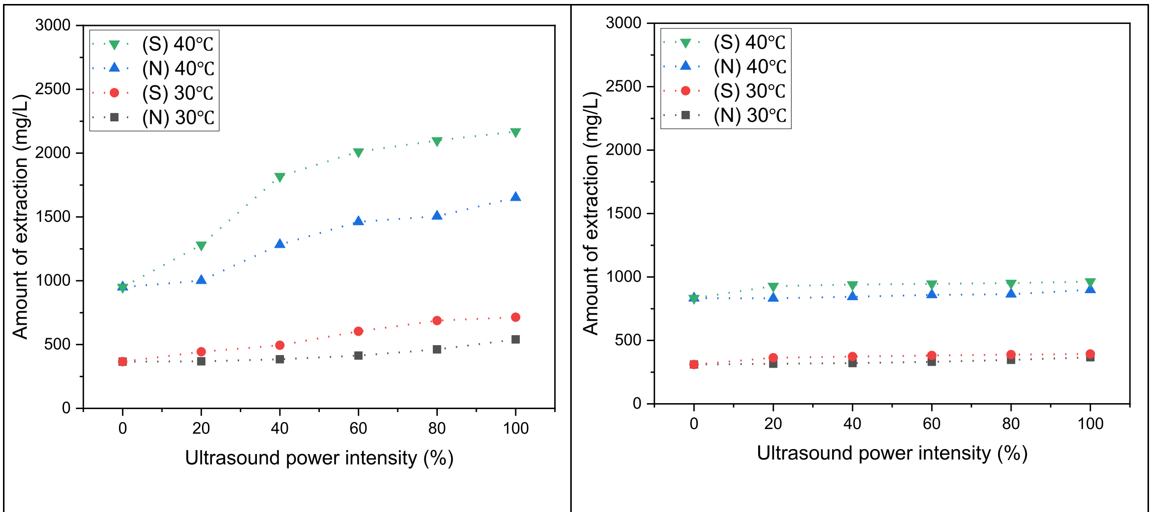
between the bubble radius and other variables in the liquid medium with equation (2) [56].

$$r = \frac{P_a}{\rho R_0\left(\omega_r^2 - \omega_a^2\right)}\left(\sin\omega_a t - \frac{\omega_a}{\omega_r}\sin\omega_r t\right) \quad (2)$$

P_a is the acoustic pressure amplitude of an acoustic wave traveling across the liquid medium, ρ is the surrounding medium density, R_0 is the bubble radius at equilibrium, ω_r is the resonance frequency of the bubble, and ω_a is the equal applied circular frequency of sound. In the above equation, besides other constant parameters of a specific liquid medium, the bubble radius r changes depending on the value of ($\omega_r - \omega_a$). The smaller the value of ($\omega_r - \omega_a$), the more bubbles are inclined to resonate. Thus, facilitating and intensifying the acoustic cavitation and collapses. As a result, the water temperature outside the column increases and facilitates the extraction of dyes. In addition, higher ultrasonic frequency generates higher energy that aids in dye extraction capabilities within the PEEK and SS columns. Whereas lower ultrasonic frequencies lead to less violent collapse of bubbles due to larger cavity size [57]. On the other hand, higher ultrasonic frequencies can lead to smaller radius of bubbles and insufficient time for cavitation bubbles to collapse, and thus reduced cavitation effect on extraction efficiency [54]. Therefore, optimizing ultrasonic frequency for best extraction efficiency is crucial for achieving highest dye extraction yield, which was 132 kHz for our study's operating conditions.

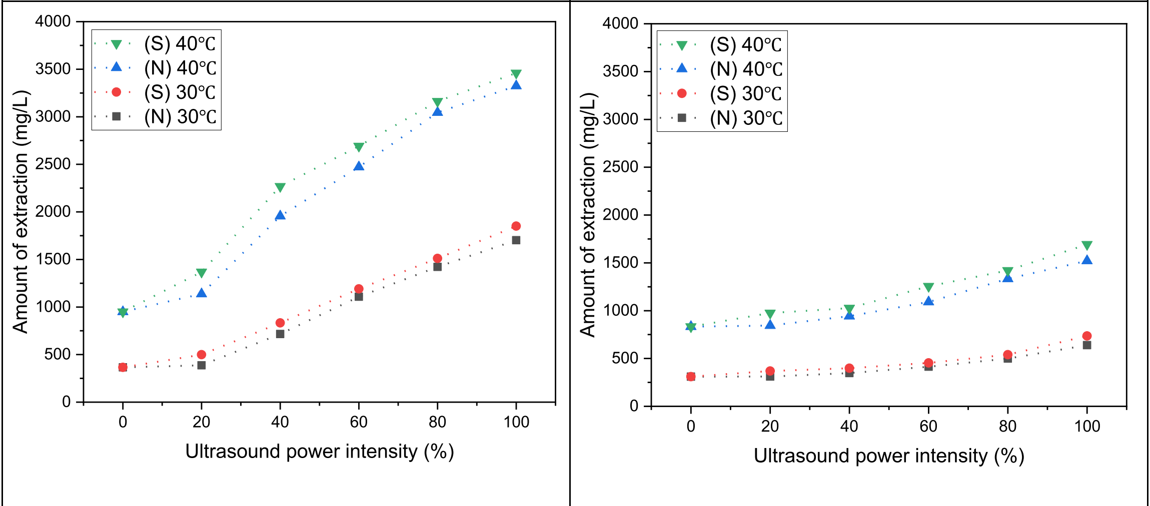
The extraction amounts between PEEK and SS column materials at different ultrasound frequencies, initial temperatures, and 100 % intensity are expressed in ratios in Table 4.

In this study, the ratio of extraction amount of PEEK material compared to that of SS material was 1.8 to 3.9 depending on the initial temperature, ultrasound frequency, and temperature control. Regardless of the initial temperature, the ultrasound frequency of 132 kHz yielded the highest extraction efficiency ratio of PEEK to SS materials. PEEK material demonstrated significantly better extraction effectiveness compared to that of SS material in the order of 132 kHz, 40 kHz, and



a. PEEK controlled temp.

b. SS controlled temp.



c. PEEK uncontrolled temp.

d. SS uncontrolled temp.

Fig. 6. Comparison of extraction effectiveness between normal (N) and sweep (S) operation modes using PEEK and SS columns with and without temperature control. (a) PEEK controlled temperature, (b) SS controlled temperature, (c) PEEK uncontrolled temperature, and (d) SS uncontrolled temperature.

Table 5
The temperature increments of bath water after the extraction using the normal and sweep operation modes[a]

Ultrasound power intensity (%)	30 °C		40 °C	
	Normal	Sweep	Normal	Sweep
20	+0.3	+0.8	0	0
40	+3.5	+4.0	+1.7	+2
60	+5.6	+6.0	+4.0	+4.2
80	+8.0	+8.7	+6.5	+7.0
100	+11.0	+11.4	+9.0	+9.7

[a] Extraction time: 30 min.

25 kHz ultrasound frequencies.

3.6. Comparison of extraction effectiveness between different operation modes

Different operation modes have different effects on the extraction process for PEEK and SS column materials. To elucidate this difference, normal (N) and sweep (S) modes of 25 kHz ultrasonic cleaning bath were tested for a comparative assessment of their effects on the extraction process. The results are shown in Fig. 6.

For both PEEK and SS columns, the 40 °C initial temperature was the most contributing factor to the highest extraction yield for all experimental conditions. The S mode further enhanced the extraction efficiencies of PEEK and SS columns compared to that of N mode. In addition, the PEEK column consistently proved to be the better material for UAE.

Another factor responsible for this observation was the acoustic standing wave. For the ultrasonic cleaning bath system, a transducer emits a single frequency wave that is transmitted upward into the bath water [56]. When the wave hits the water surface, it is reflected downwards causing a standing wave. The standing wave only produces fixed cavitation zones or immobilizing energy at certain points along the water depth corresponding to a half of wavelength of the frequency used. To reduce or avoid this standing wave, manufacturers assembled the ultrasonic generator with a circuit to slightly vary the emitted frequency over time known as the frequency sweep. Thereby, the energy can be distributed more evenly with the S mode. Furthermore, some ultrasonicators do not provide N and S modes and rather have a fixed preset of S mode only. To examine the N and S modes' effect on bath temperature, the temperature increments under uncontrolled temperature conditions were recorded in Table 5.

The S mode resulted in slightly higher temperature increments than that of the N mode. These results agree with the mechanisms discussed in Sections 3.2 and 3.3. The S mode facilitated the production of more bubbles and collapse due to small changes in frequency from low to high and vice versa. Therefore, the water temperature increment for S mode was greater than that of N mode. Consequently, more dye particles were extracted from the fabric inside the columns. It can be concluded that the S mode of sonication can accelerate the extraction process better than the N mode.

4. Conclusion

In this study, comparison of the effects of ultrasound intensity, initial bath temperature, ultrasound frequency, and operation modes on the extraction efficiency of PEEK and SS column materials under controlled and uncontrolled temperature conditions was conducted. Our study demonstrated that the PEEK material was more significantly affected by the aforementioned factors than the SS material under identical conditions, thus exhibiting enhanced extraction effectiveness especially via S mode operation. At the maximum ultrasound intensity, the extraction effectiveness ratio of PEEK to SS columns was in the ranges of 1.8 – 3.9 depending on the specific ultrasound frequency and initial temperature condition. In terms of enhancing extraction effectiveness, the optimum ultrasound frequencies are arranged in order of best to worst: 132 kHz, 40 kHz, and 25 kHz. Unlike SS material, the extraction efficiency of PEEK material was more affected by temperature and acoustic effects especially at 132 kHz ultrasound frequency. At lower ultrasound frequencies, the acoustic effect was insignificant for both column materials when the bath temperature was controlled. Based on obtained data, the acoustic effect did not have significant effect on the SS column with respect to UAE of dye when compared to that of PEEK column. The comparison of the extraction efficiencies between the SS and PEEK columns in UAE under various ultrasonic conditions determined that higher extraction efficiency was correlated with the PEEK material at the ultrasound frequency of 132 kHz, which helped explicate the underlying mechanism of the UAC. Conclusively, although the mechanisms of ultrasound in chiral separation or ion-exchange require further investigation, the comparative assessment of UAE in this study can explain how the properties of different column materials contribute to the UAC results observed in the previous studies [23–27] using the SS column in the chiral chromatography and PTFE (or PEEK) column in the ion chromatography.

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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