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Butane extraction of model organic pollutants from water

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Abstract

In this study, n-butane (n-C_4H_{10}), a by-product of the oil refining process, was used as the extractant to remove various model organic pollutants including halogenated hydrocarbons, phenols and aromatic compounds from aqueous matrices. The presence of salt, inorganic acid and dissolved organic materials in the aqueous matrix were found to have little influence on the removal efficiency. High removal efficiencies are readily achievable for a great number of organic pollutants. The removal efficiencies for hydrophobic pollutants were greater than 90% for a single stripping stage for pollutants with a distribution constants (K_D) greater than 45 and for a n-butane to aqueous phase ratio of 1–5. Results were also reported for the removal of residual butane in treated effluent by combinations of depressurization, air stripping and elevating operating temperature.
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Keywords: n-Butane; Extraction; Organic pollutants; Water; Distribution constant

1. Introduction

Contamination of aquatic systems by organic compounds is one of the most important pollution problems in both industrialized nations and developing nations. Even though wastewater treatment technologies have been improved greatly over the last three decades, water pollution is an evolving problem with the rapid developing industrial and ever-expanding agricultural activities. Many current technologies were outmoded or need substantial modification to meet the new environmental challenges. Moreover, owing to the nature of pollution varies from case to case, no single technology is able to treat the whole spectrum of pollutants and the complications of wastewater. This has stimulated new approaches in alternative and innovative technologies to clean up wastewater in an effective, timely and economical way.

Based on traditional solvent extraction techniques, there is great interest in using liquid butane or similar highly volatile non-polar solvents as an alternative to conventional solvents to remove organic compounds from various ma-

trices. When gases such as propane and butane are liquefied, they have physicochemical properties that are ideal and notably non-toxic extractants. In recent years, liquefied gases have been applied to the removal of pollutants such as polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) from environmental matrices including soils, sediments and sludges [1–3]. Chriswell and Schmitt [4] reported that high recoveries were obtained within 1 h when liquid butane and trimethylpentane were used to treat a high-humus soil and a sandy soil sample artificially contaminated with toluene or gasoline in a concentration range of 0.1 and 1.0 μg/g, respectively. Teo and co-workers [5–8] reported that the butane extraction process is capable of effective removal of toxic organic pollutants from water. They reported that various model organic compounds spiked into water were tested on a small scale, single stage, bench-type unit as well as a continuous counterflow 201 pilot-plant with four mixing and extraction chambers.

From these studies [1–8], it can be concluded that butane extraction offers a promising alternative to existing technologies for environmental cleanup. There exists, however, no systemic study of the method in detail. We have, to date, only a basic understanding of the butane extraction process. The present study is focused on developing a larger database for better understanding of this technology.

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Fundamental issues such as partition coefficients, effects of salts and surfactants are explored. The experimental results of the present work may provide the physical and chemical basis for the future design of a continuous pilot-scale butane extraction system. Model organic compounds were selected to mimic water pollutants, involving both common industrial chemicals and toxins of special interest. Model compounds included halogenated compounds, aromatics and phenolics on the US EPA priority list for water pollutants.

2. Materials and methods

2.1. Reagents

All chemicals used in this study were obtained from Aldrich Chemicals Co., USA and were of highest purity available. Normal butane with purity greater than 99% was supplied by Messer Singapore Pte. Ltd. Reagent grade water was prepared by passing doubly distilled water through an ultra-pure water system (Milli-Q plus 185, Millipore, USA). The reagent-grade water contained no measurable organic background concentrations of any target analytes under the experimental conditions. Stock aqueous solutions were prepared by spiking the appropriate amount of the pure chemicals into reagent water and were homogenized using an ultrasound bath.

2.2. Instrument

The basic laboratory-scale processing unit was designed and constructed in-house [7]. The device comprises a thick-wall Pyrex glass extractor unit with an inner diameter of 80 mm, a height of 160 mm and a wall thickness of 8 mm. Four internal vertical stationary baffles were evenly spaced along the inner wall to provide maximum turbulence mixing to prevent the vortex formation in order to enhance the extraction rate. The extractor was designed to withstand pressures (cf. 150–250 kPa) that exceed normal atmospheric pressure.

The 1028S iso-temperature refrigerated circulators from Fisher Scientific, Inc., USA was used to control the operating temperature. Laboratory stirrer was supplied by Kika Works (Asia) Sdn. Bhd., Malaysia. A 12-channel scanning thermocouple thermometer was obtained from Colo-Parmer Instrument Co., USA.

2.3. Test procedures

The experiments were performed for the chosen model organic compounds spiked into water. n-Butane (n-C_4H_{10}) was used as an extractant to remove these organic compounds at the optimized operating conditions listed in Table 1. Freshly prepared aqueous stock solution of the model organic compounds was transferred into the extractor at a predetermined volumetric ratio (n-butane/water = 1/5). Liquid butane was supplied by means of condensation or by placing the butane tank in reverse direction for direct liquid transfer. The operating temperature was maintained at 298 K by a thermostated water-bath. The two-phase solution was agitated at a given speed ranging from 300 to 1000 rpm. Each experiment lasted till extraction equilibrium was established, usually less than 30 min, and samples were taken periodically for every 0.5 min for the initial 2-min period of the experiment, thereafter, every 5 min per sampling. Samples were drawn from aqueous phase via a long (150 mm) 316 stainless steel syringe needle. Complete phase separation was readily achieved under normal gravity within a few minutes. The experiments were all conducted in triplicate.

Halogenated compounds in water samples were analyzed by Tekmar 2016/3000 purge-and-trap (P&T) concentrator coupled with Varian 3600 gas chromatograph (GC) equipped with electron capture detector (ECD) and flame ionization detector (FID). Aromatic compounds and phenolic compounds were analyzed by Varian HPLC equipped with Varian 9050Q solvent delivery system, Varian 9100 autosampler and 9012 variable wavelength UV-Vis detector. The concentrations of the analytes were determined with reference to external standards. The removal efficiencies (R) were obtained, and the distribution constants (K_D) were derived from R values and the volumes of the two-phases [5–8]. By definition, the distribution constant (K_D) expresses the equilibrium concentration ratio of an organic solute partitioned between an organic phase and the aqueous phase.

3. Results and discussion

3.1. Removal of model organic compounds from water with n-butane

A variety of model organic compounds were spiked in reagent water at the concentrations ranging from 70 to 1200 mg/l. As shown in Table 2, removal efficiencies of a

Table 1
Operation conditions of butane extraction of model compounds from water

Aqueous feed	300 ml	Solute concentration	70–1200 mg/l
Liquid n-butane	60 ml	Condensing temperature	268 K
pH adjustment	No	Surfactant concentration	Nil
Stirring speed	300–1000 rpm	Pressure	300–500 kPa
Extraction temperature	298 K	Extraction time	30 min

Table 2
Results of single stage butane extraction of model organic compounds from water

Model compounds	C_0 (mg/l)[a]	R (%)	K_D	Model compounds	C_0 (mg/l)[a]	R (%)	K_D
Chloroform	269.8	90.3 ± 3.6[b]	46.5 ± 1.9	Nitrobenzene	240.7	82.4 ± 1.1	23.4 ± 0.3
Bromodichloromethane	110.5	91.9 ± 3.3	56.7 ± 2.0	Chlorobenzene	329.3	99.3 ± 2.5	709 ± 17.9
Bromoform	595.9	94.4 ± 1.8	84.3 ± 1.6	o-Dichlorobenzene	156.0	99.5 ± 4.3	995 ± 43.0
Carbon tetrachloride	200.0	97.8 ± 7.6	222 ± 17.0	p-Dichlorobenzene	76.0	99.8 ± 5.4	2495 ± 135
1,2-Dichloroethane	672.9	81.3 ± 3.4	21.7 ± 0.9	Phenol	105.4	2.6 ± 0.3	0.13 ± 0.02
1,2-Dibromoethane	1207	92.2 ± 2.3	59.1 ± 1.5	m-Cresol	88.8	7.2 ± 0.6	0.39 ± 0.03
1,1,1-Trichloroethane	67.7	97.6 ± 0.6	203 ± 1.3	p-Cresol	97.8	11.2 ± 0.6	0.63 ± 0.03
Trichloroethylene	459.0	98.8 ± 0.7	412 ± 2.9	o-Nitrophenol	99.1	81.4 ± 7.0	21.9 ± 1.9
1,1,2,2-Tetrachloroethane tetrachloroethane tetrachloroethane	112.5	88.0 ± 2.6	36.7 ± 1.1	m-Nitrophenol	108.7	26.8 ± 1.2	1.83 ± 0.08
Perchloroethylene	108.4	98.3 ± 1.3	289 ± 3.8	p-Nitrophenol	138.0	26.9 ± 1.4	1.84 ± 0.10
1,2-Dichloropropane	696.0	90.0 ± 1.2	45.0 ± 0.6	o-Chlorophenol	88.2	42.4 ± 2.9	3.68 ± 0.25
1,2,3-Trichloropropane	344.9	92.4 ± 1.7	60.8 ± 1.1	p-Chlorophenol	84.7	7.2 ± 0.8	0.39 ± 0.04
Trichloroacetonitrile	257.7	98.0 ± 1.2	245 ± 3.0	o-Bromophenol	210.2	55.0 ± 1.7	6.11 ± 0.19
Benzene	935.6	97.4 ± 6.6	187 ± 12.7	1-Naphthol	500.0	27.4 ± 1.9	1.89 ± 0.13
Toluene	400.0	99.1 ± 2.9	551 ± 16.1	n-Butyl acetate	898.1	90.0 ± 1.8	45.0 ± 0.9
o-Xylene	150.0	99.7 ± 4.0	1661 ± 67.0	Methanol	1662	≈0	NA
Ethylbenzene	112.5	99.9 ± 2.0	4995 ± 100	Acetonitrile	1000	≈0	NA

[a] C_0 represents initial concentration of model pollutant spiked in water.
[b] The value after ± is standard deviation, $N = 3$.

single stage n-butane extraction range from a few percent up to 100% for the studied model compounds. The values of K_D derived from R values based on an n-butane to aqueous phase ratio of 1:5 ranged from 10^{-1} to 10^5. The results demonstrate that high removal efficiencies ($R \geq 95\%$) were achieved for hydrophobic halogenated hydrocarbons and aromatic compounds in a single stage extraction, whereas lower efficiencies were achieved for hydrophilic compounds such as methanol, acetonitrile and majority of phenolic compounds (with the exception of approximately 80% removal for o-nitrophenol). Clearly, n-butane is a less useful solvent for the removal of polar phenolic and water-miscible compounds from water due to its relatively low polarity.

Although the quality of treated water may not meet the required sanitation standard in a single batch extraction, the technique has promise for use in a multiple stage extraction in the continuous treatment facility. Therefore, a continuous operating system test should be investigated further.

3.2. Effect of temperature on butane extraction

Temperature can have a considerable impact on the overall butane extraction efficiency strongly related to the equilibrium constants, kinetics and phase separation. It has been established that aqueous solubility of the organic pollutants has a dominant effect on the efficiency of butane extraction [5,8]. Solubility of a solute in water is a function of temperature, but the exact potency and effect can be greatly variable. Many organic compounds become more soluble as the temperature increases, but some behave in an opposite manner. Solubility for some compounds might either increase or decrease at higher temperatures, depending on their nature and the temperature range involved. Therefore, it is

essential to investigate the effect of temperature on butane extraction.

Table 3 collects the experimental values of n-butane/water distribution constant for the chosen model compounds measured at operating temperatures ranging from 278 to 308 K (data at 298 K, result given in Table 2). Based on these results, the following linear regression equation was performed for each model compounds:

$$\log K_D = A_1 \times T + B_1 \tag{1}$$

where K_D is the n-butane/water distribution constant, T the temperature in K and A_1, B_1 are the constants, respectively. The slope A_1 and intercept B_1 derived for these compounds are listed in Table 3. Given the limited data collected, the linearity of the solubilities as a function of temperature are surprisingly good with correlation coefficients (r^2) values ranging from 0.87 to 0.96. As can been seen from Table 3, the $\log K_D$ generally has a small dependence on temperature (the A_1 column of Table 3) varying from 0.001 to 0.02 per degree K. This agrees well with the results reported Leo et al. [9]. Some broad observations can be made of the variation of K_D with temperature based on the chemical identifies of the solutes. In general, the halogenated compounds have an inverse relationship with temperature, increasingly partitioning to the aqueous phase. In contrast, the aromatic and the phenolic compounds partition more completely into the n-butane phase with increasing temperature.

It can be concluded from Table 3 that the extraction temperature will have rather minor impact on the K_D values, and subsequently on the removal efficiency. Furthermore, higher temperatures lead to higher operating pressures, and it will result in higher costs for the extraction operating system by requiring more stringent system design and increased

Table 3
Distribution constants of butane extraction and log K_D as a function of temperature

Compounds	Temperature (K)				$\log K_D = A_1 \times T + B_1$		
	278	288	298	308	A_1	B_1	r^2
Chloroform	50.5	46.8	46.5	45.0	−0.0016	2.13	0.875
Carbon tetrachloride	431	341	222	197	−0.0120	5.97	0.964
Bromodichloromethane	66.0	58.7	56.7	51.3	−0.0034	2.77	0.962
Perchloroethylene	559	441	289	163	−0.0179	7.76	0.968
Bromoform	96.9	86.7	84.3	63.4	−0.0056	3.56	0.863
Benzene	121	126	187	192	0.0074	0.02	0.859
Nitrobenzene	20.6	22.3	23.4	23.5	0.0019	0.78	0.886
o-Nitrophenol	20.4	21.6	21.9	22.3	0.0012	0.98	0.890
o-Bromophenol	5.18	6.10	6.11	6.84	0.0036	−0.28	0.862

energy demand. Therefore, it seems prudent to perform the extraction at near ambient temperature, i.e. 293–303 K.

3.3. Effect of salinity and acidity on butane extraction

In many cases, it may be necessary to consider the chemical conditions of the aqueous matrix such as acidity and salinity for some wastewater treatment using butane extraction technology.

Hydrogen ion concentration could possibly affect butane extraction via the changing of the solubility of organic compounds in water. Organic acids will be expected to increase in aqueous solubility with increasing pH, while organic bases should behave in the opposite way. Even the solubility of "neutral" organic compound (e.g. alkanes and chlorinated hydrocarbons) can be affected by the pH. Hence, pH adjustment might be necessary for some extractions in order to achieve the most favourable results in terms of removal efficiency.

The wastewater to be treated can also be of high salinity, in the case of, for example, contaminated seawater. There is often concern as to whether the extraction model is applicable to such a situation and what potential difficulties increased salinity might pose for the extraction efficiency. In general, the presence of dissolved salts or minerals in water leads to a decrease in organic solute solubility. The extent of this "salting out" varies considerably with different so-

lutes and salts, and can only be determined experimentally. For example, the solubilities of several polycyclic aromatic hydrocarbon compounds in seawater, which contains about 35 g/l sodium chloride (NaCl), are reduced by 30–60% below their pure water solubilities. On the other hand, the solubility of short chain n-alkanes in salt water as compared to that in fresh and distilled water is greater by one order of magnitude. This difference decreases with an increase in the molecular weight of the hydrocarbon [10].

Distribution constants for n-butane extractions are shown in Table 4 for solutions acidified with HCl acid to a pH value of 2.0. It was found that there are marked increases of the distribution constant in the case of the acidified aqueous solutions as compared to the K_D values without adjustment at 298 K. Because n-butane is a neutral liquid, the pH of the aqueous phase has little effect on its extraction characteristics. The variations in extraction efficiencies for the individual solutes may be explained by considering their acid/base character. Phenol and its derivatives are weak Lewis organic acid. Aqueous solutions of halogenated hydrocarbon often present as weak acids, hydrolysing to form hydrogen halide such as HCl and HBr (see Table 4). Increasing the acidity of the aqueous phase reduce the solubilities of these organic species in water, thus increasing the n-butane/water distribution constants for these compounds.

NaCl was also added to the aqueous solution to maintain concentrations of 0.1 and 0.4 M to simulate the salinity

Table 4
Effect of acidity and salinity on the distribution constant of butane extraction

Compounds	pH[a]	pH adjustment		Salinity adjustment (M NaCl)	
		Without pH adjustment	pH 2.0	0.1	0.4
Chloroform	6.2	46.5	151	67.5	77.0
Carbon tetrachloride	6.7	222	1245	450	709
Bromodichloromethane	6.7	56.7	203	79.7	85.9
Perchloroethylene	6.8	289	328	308	328
Bromoform	6.7	84.3	222	99.2	127
Benzene	7.0	187	NA	109	117
Nitrobenzene	5.0	23.4	23.6	23.2	22.9
Phenol	4.5	0.13	0.35	0.13	0.13
o-Nitrophenol	4.7	21.9	22.6	22.6	22.9

[a] Measured pH value of aqueous solution without adjustment of acidity.

Table 5
Distribution constants of butane extraction and $\log K_D$ as a function of methanol content in water

Compounds	Methanol concentration (vol.%)			$\log K_D = A_2 \times C + B_2$		
	0	1.0	5.0	A_2	B_2	r^2
Chloroform	46.5	44.5	40.9	−0.009	1.58	0.970
Carbon tetrachloride	222	142	66.4	−0.099	2.31	0.968
Bromodichloromethane	56.7	42.6	32.6	−0.050	1.81	0.867
Perchloroethylene	289	212	95.0	−0.164	2.90	0.961
Bromoform	84.3	38.8	34.4	−0.014	1.74	0.983
Trichloroethylene	412	273	233	−0.043	2.56	0.719
Chlorobenzene	709	620	551	−0.021	2.84	0.830
Phenol	0.13	0.12	0.12	−0.001	−0.89	0.774
m-Cresol	0.39	0.36	0.36	−0.006	−0.42	0.745
o-Chlorophenol	3.68	3.53	3.36	−0.007	0.56	0.920
p-Chlorophenol	0.39	0.36	0.34	−0.010	−0.42	0.744
o-Nitrophenol	21.9	19.4	17.9	−0.014	1.30	0.828

conditions similar to that of diluted seawater and seawater, respectively. The effect of salinity on distribution constants of butane extraction is given in Table 4. It can be seen that salinity has less influence than acidity for butane extraction for most of the model compounds. Overall, distribution constant increases as the salinity increases; however, the magnitude of increment is not significant for the majority of the organic solutes.

3.4. Effect of dissolved methanol on butane extraction

The presence of dissolved organic material such as proteins, humic and fulvic acids can lead to an increase in the solubility of many organic compounds in water and potentially can affect the removal efficiency by solvent extraction. Table 5 summarizes the results on the distribution constants of butane extraction for various model compounds in the presence of 1 and 5% of methanol in the aqueous phase.

In order to evaluate the effect of methanol on butane extraction of other trace pollutants, the logarithmic values of experimental distribution constants were plotted against the methanol concentrations in the aqueous phase. The plot can be linearized and the regression relationship can be expressed as $\log K_D = A_2 \times C + B_2$.

Values of the slope (A_2) were listed in Table 5 along with the values of intercept (B_2) and squares of correlation coefficients (r^2). It is shown that there are small decreases in distribution coefficient in the presence of methanol in the order of 0.001–0.16 of the $\log K_D$ units per volume percent of methanol present in the aqueous phase. Overall, the negative effect of methanol in water, is therefore, proven to be less significant than as expected.

3.5. Effect of surfactant on butane extraction

The use of aqueous surfactant has been a common practice to wash contaminated soil and objects. In this study, butane extraction was used to remove organic pollutants from the "water-wash-out" wastewater effluent. The model surfactant used in this study was Triton X-114, a general purpose type nonionic liquid detergent, which is commonly used in the formulation of industrial detergents and present in emulsifiers [11].

Comparing the results shown in Table 6, it was found that the effect of surfactant in water is not very clear. The presence of 1% Triton X-114 led to an overall enhancement on the distribution constant for most of the studied model compounds. However, the presence of this surfactant could lead to formation of stable emulsions between butane and aqueous phases slowing down the rate of phase separation. The persistent emulsion problem could not be removed by usual means of either centrifuging or gravity phase separation. Efforts including changing operating temperature and the salinity, have been applied to improve the process, unfortunately, none were effective. From this preliminary study, it is clear that surfactant in the feed water can cause significant problems for butane extraction. Therefore, it is recommended that one should avoid the use of surfactant in the pre-treatment process of wastewater if butane extraction is designated to be the remediation process.

Table 6
Effect of concentration of surfactant on the distribution constants of butane extraction

Compounds	Triton X-114 concentration (vol.%)		
	0	0.1	1.0
Chloroform	46.5	37.0	106
Carbon tetrachloride	222	53.8	620
Bromodichloromethane	56.7	31.0	66.4
Perchloroethylene	289	47.1	828
Bromoform	84.3	64.4	308
Phenol	0.13	0.33	0.92
m-Cresol	0.39	0.69	1.27
o-Chlorophenol	3.68	4.06	4.45
p-Chlorophenol	0.39	0.95	1.43
o-Nitrophenol	21.9	18.0	13.1

Table 7
Air-stripping of residual butane in treated water (mg/l) as a function of air flow rate and temperature

Time (min)	Air flow rate (ml/min)[a]				Temperature (K)[b]		
	50	100	200	300	288	298	308
0	53.2	53.3	52.9	52.4	53.3	53.3	53.3
2	22.7	6.9	3.9	1.1	8.9	6.9	5.8
5	7.7	1.5	1.0	0.5	1.7	1.5	1.2
10	2.4	0.9	0.9	0.5	1.1	0.9	0.7
20	2.2	0.7	0.3	0.1	0.8	0.7	0.5

[a] Operated at 298 ± 5 K.
[b] Air flow rate at 100 ml/min.

3.6. Removal of residual butane in treated water

In the design of an extraction system, one should not overlook that butane might be taken up by the aqueous phase and thus further contaminate the treated water. Organic dissolution and entrainment in aqueous phase is inevitable, even though with efforts made to minimize it. In addition, certain organic pollutants possibly might have some positive effect on the butane solubility, the so-called "salting-in" effect, which raised concern on the loss of extractant and the effect of entrained butane on the environment. Therefore, effort must be taken to reduce the butane concentration to an acceptable level before the treated water can be discharged to any receiving water body.

In principal, the residual butane in water might be driven off by means of depressurization and/or in combination with a higher operation temperature. In this study, treated water containing residual butane as described in Section 2 was first placed in a vessel to depressurize to atmospheric pressure at ambient temperature (293–303 K) without air stripping. The depressurized aqueous phase were transferred to a cylindrical air striping tower (25 mm × 300 mm, i.d.) and sparged with air stream under atmospheric pressure, the air dispersed into the aqueous matrix via a fritted dispersion glass tube.

As can be seen from Table 7, air stripping at atmospheric pressure can rapidly reduce the dissolved butane to less than 0.1 mg/l. This compares favourably with the saturated aqueous solubility of 61.2 mg/l at room temperature [12]. Higher air flow rate increases the mass transfer of the dissolved n-butane to the gas phase and reduces the sparging time. An air flow rate of 100 ml/min required more than 10 min while a flow rate of 500 ml/min only needed 5 min in order to reduce residual butane from 50 mg/l to less than 1.0 mg/l.

The effect of temperature on air stripping of residual butane was also reported in Table 7. It can be seen that higher temperature reduces the butane solubility in water, but the effect is not very significant over the temperature range tested. At 298 K, under an air flow rate of 100 ml/min, the butane concentration in water can be readily reduced by 98.6% from 53.3 to 0.7 mg/l.

From these results, at a temperature 288 K or higher, and under an air flow rate of 100–300 ml/min with an appro-

priate air stripping device, the residual butane can be controlled to less than 1.0 mg/l in the aqueous phase. Such low levels of residual n-butane needs not be of great concern. For example, n-butane is often used as an extractant for the refinement of animal grease and vegetable oil. It is also approved by the Food and Drug Administration of the USA (US FDA) as a general-purpose food additive. Therefore, a small residual concentration of n-butane remaining in the treated water should not pose any threat to the environment and living organisms.

4. Conclusions

Laboratory experimental results have shown that butane extraction has the potential to remove a broad range of model organics from aqueous phase and is particularly effective for hydrophobic organics. The method can offer high removal efficiencies in a single extraction stage with n-butane to aqueous phase volume ratio of 1:5 for the chosen model organic compounds except for phenols and more water-miscible compounds. Tentative efforts were made to remove the residual butane from treated water by depressurization and air stripping processes.

The effect of various factors on butane extraction were discussed in this study. It was found that the presence of salt, acid and dissolved organic materials in aqueous matrix has little influence to the overall extraction performance. However, if the matrix contains excessive high concentration of these materials, butane extraction may be not as effective as expected. The presence of detergents or emulsifiers can have negative impact on the extraction performance in terms of time required for breaking the emulsion to achieve the desired phase separation.

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