Effervescence-Assisted Microextraction

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Effervescence-assisted microextraction emerged in 2011 as a new alternative in this context. The technique uses in situ-generated carbon dioxide as the disperser, and it has been successfully applied in the solid-phase and liquid-phase microextraction fields. This minireview explains the main fundamentals of the technique, its potential and the main developments reported.

dispersion micro-solid phase extraction

1. Introduction

The contact surface area between the donor (sample) and the acceptor (extractant) is a critical variable in the context of microextraction techniques [1]. The efficient dispersion of the extractant phase into the sample in the form of a fine suspension of micro/nano particles or drops is the most common approach to enhance the close contact between phases [2]. Several strategies, both in the solid-phase and liquid-phase microextraction contexts, have been proposed. These strategies can be generally divided into two main groups depending on the use of a external energy source [3][4] or chemicals [5][6] to achieve this dispersion.

In 2011, a new dispersion strategy was proposed by Lasarte-Aragonés et al. [7] and, since then, it has been developed and applied by many different groups worldwide. The technique is based on the *in situ* generation of carbon dioxide because of the reaction between a carbon dioxide donor and a proton donor in the so-called effervescent reaction. The CO_2 bubbles generated produces an efficient dispersion of the extractant phase into the sample. The technique has been successfully applied to the solid-phase and liquid-phase microextraction contexts, as a green, cheap, and simple alternative.

2. Effervescence-Assisted Dispersive Micro-Solid Phase Extraction

Dispersive solid phase extraction (DSPE) is based on the dispersion of a solid sorbent into the sample of interest proposed by Anastassiades [8] as clean-up method aimed to remove interferences from the matrix, aided by vortex agitation. This kind of cleanup strategy later received the name of QuEChERS, an acronym for its advantages: quick, easy, cheap, effective, rugged, and safe and is now commercially available as a sample treatment strategy [9].

The miniaturized version is usually named dispersive micro-solid phase extraction (DμSPE). The cornerstone of DμSPE is the dispersion of the sorbent (extractant phase) in the sample. The process must take into consideration the nature and properties of the sorbent (polarity, micro or nano-size, aggregation) and can be achieved by physical or chemical means. Physical dispersion is typically assisted by an external energy source such as ultrasound irradiation [10] or vortex agitation [11]. Chemical dispersion is aimed to improve dispersibility by using a water-miscible organic solvent such as acetonitrile or methanol [12].

The first effervescent-based approach for this alternative is based on the fabrication of a tablet containing a commercial sorbent (OASIS-HLB) and reaction precursors (sodium carbonate as CO_2 source and sodium dihydrogen phosphate as proton donor) [7]. The tablet containing all the elements was then introduced into the aqueous sample, and the sorbent is effectively dispersed by the in situ-generated gas bubbles. The alternative is designed to provide all the elements to perform the extraction process on-site, avoiding the use of a disperser solvent (minimizing environmental impact and waste generation) or external apparatus (such as vortex or ultrasounds). In fact, the first implementation of the technique employs a syringe as both sample-collection and extraction vessel device. The effervescent sorbent tablet is placed inside the syringe and the extraction process starts once the sample is aspirated. The dispersed sorbent can be easily recovered by a syringe filter and eluted before analysis. The method was employed for the determination of nitroaromatic compounds in water samples. Its analytical performance was comparable to other SPE alternatives for the same analytical problem, but in a simpler and rapid fashion. Analyte partition equilibrium is not affected by the effervescence process, and different sorbents can be used according to the target analytes.

The potential of effervescent tablets was later demonstrated in the effective dispersion of a nanometric sorbent, multiwalled carbon nanotubes (MWCNTs), in aqueous matrices [13], which are known for their limited dispersibility due to their trend to aggregation. The use of effervescent tablet (102 mm ID) was responsible for the efficient dispersion of the unmodified sorbent, resulting in more effective than mechanical agitation. By this means, a small amount of sorbent (7.5 mg) can be successfully dispersed in a large sample volume (100 mL) without the assistance of any external apparatus or energy source. The proposed alternative combined with Liquid Chromatography-Diode Array Detector (LC-DAD)

3. Effervescence-Assisted Dispersive Liquid-Phase Microextraction

Dispersive liquid–liquid extraction (DLLME), proposed by Rezaee et al. in 2006, is based on the efficient dispersion of an extractant solvent into the sample [14]. In the typical approach, this dispersion is aided by a disperser solvent that is miscible with both the sample and the extractant phase, which is recovered by centrifugation. The use of an organic solvent as the disperser has two main shortcomings. On the one hand, the volume of disperser solvent is in the mL range, and, therefore, DLLME cannot be completely considered a microextraction technique. On the other hand, the disperser solvent is mixed with the aqueous sample, increasing the analytes' solubility in the donor phase, reducing its transfer to the extractant phase. The need for a disperser solvent can be reduced if an external energy source, like US [36] and vortex [5], is used.

Lasarte-Aragones et al. proposed the adaptation of effervescence extraction to the LPME context in 2014 [15]. Effervescence-assisted DLLME (EA-DLLME) consists of the in situ production of carbon dioxide due to the reaction of sodium carbonate (previously added to the sample) and the disperser solvent (acetic acid), which also contains the extractant phased. The reaction also generates sodium acetate that contributes to the ionic strength and may produce a salting-out effect. In this preliminary work, magnetic nanoparticles were added to the acetic acid-extractant mixture and used to recover the extractant from the sample avoiding the centrifugation process. The extractant solvent, 1-octanol, is recovered by the interaction of the alcohol group with residual hydroxyl groups on the surface of the nanoparticles.

4. Developments of Effervescence-assisted microextraction

Since its introduction, many researchers have expanded the applications of the concept to a variety of sorbents and solvents in different matrices, demonstrating the wide range of applicability of the technique. A detailed and complete review of these methodologies can be found in the recent publication by our research group [16]. The main contributions to the technique are presented in Table 1.

Table 1. Applications based on the use of effervescence-assisted microextraction.

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
Na_2CO_3	NaH_2PO_4	Oasis HLB	Water	10 mL	Nitroaromatic compounds	The tablet is placed on the syringe used as extraction vessel. Effervescence occurs upon sample aspiration inside the vessel. Sorbent with extracted analytes is recovered by syringe filter.	[7]
Na_2CO_3	NaH_2PO_4	MWCNTs	Water	100 mL	Triazines	Nanotubes are only effectively dispersed in tablet format with no	[13]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						additional organic solvent.	
Na_2CO_3	NaH_2PO_4	G-MWCNTs-COOH	Hawthorn herb	200 mL	Natural antioxidants	The tertiary tablet is prepared by blending the ingredients and applying pressure. Different types of nanotubes were evaluated.	[17]
Na_2CO_3	NaH_2PO_4	Mesoporous hybrid materials (PCMA-60)	Root extracts	20 mL	Tanshinones	The tertiary tablet is prepared by blending the ingredients and applying pressure. Higher amounts of sorbent (13 mg) produce aggregation and decrease in extraction efficiency	[18]
Na_2CO_3	NaH_2PO_4	β-cyclodextrin/attapulgite composite	Water	7 mL	Pyrethroids	The tablet is placed on the syringe used as extraction vessel. Effervescence occurs upon sample aspiration inside the vessel. Sorbent with	[19]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						extracted analytes is recovered by syringe filter	
Na_2CO_3	NaH_2PO_4	Magnetic attapulgite/polypyrrole nanocomposites	Honey	100 mL (diluted)	Pyrethroids	The tertiary tablet is prepared by blending the ingredients and applying pressure. Magnetic properties of the sorbent are used to facilitate sorbent recovery.	[20]
Na_2CO_3	NaH_2PO_4	IL-Magnetic-β-cyclodextrin/attapulgite composite	Honey and Juice	8 mL	Fungicides	The tertiary tablet is prepared by blending the ingredients and applying pressure. The sorbent is easily recovered using an external magnet.	[21]
Na_2CO_3	NaH_2PO_4	$NiFe_2O_4$ MNPs	Seafood extracts	30 mL	Heavy metals	The tertiary tablet is prepared by blending the ingredients and applying pressure. The sorbent is easily recovered	[22]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						using an external magnet.	
Na_2CO_3	Citric acid	Fe_3O_4/chitosan-Se MNPs	Sausage extracts and Water	10 mL	Heavy metals	The tertiary tablet is prepared by blending the ingredients and applying pressure. The sorbent is easily recovered using an external magnet. Selenium increase extraction potential for metal ions	[23]
Na_2CO_3	Citric acid	Dopamine-modified magnetic graphene oxide	Sausage extracts and Water	100 mL	Metal ions	The tertiary tablet is prepared by blending the ingredients and applying pressure. The sorbent is easily recovered using an external magnet. Dopamine enhances extraction capacity.	[24]
Na_2CO_3	Citric acid	Dopamine-carbon graphite nitride nanosheets	Oil and water samples	100 mL	Metal ions	The tertiary tablet is prepared by blending the ingredients and applying pressure. Sorbent with	[25]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						extracted ions is separated by centrifugation.	
Na_2CO_3	Citric acid	Fe_3O_4@SiO_2@N_3 MNPs	Urine and pharmaceutical wastewater	10 mL	Antidepressant drugs	The tertiary tablet is prepared by blending the ingredients and applying pressure. The sorbent is easily recovered using an external magnet. Nitrogen-rich surface increases adsorption capacity.	[26]
Na_2CO_3	Tartaric acid	Ni-based N-doped Graphene tubes	Deproteinized milk	5 mL	Bisphenols	The tertiary tablet is prepared by blending the ingredients and applying pressure. Magnetic properties of Ni-based tubes are used to facilitate sorbent recovery.	[27]
Na_2CO_3	NaH_2PO_4	Core-shell magnetic COF	Water, beverages and biosamples	5 mL	Endocrine disruptors	The tertiary tablet is prepared by blending the ingredients and applying pressure.	[28]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						Magnetic properties of the sorbent are used to facilitate sorbent recovery.	
$NaHCO_3$	NaH_2PO_4	benzo-15-crown-5	*C. fraxini* medicinal plant	-	Coumarins	The procedure consists of matrix solid-phase dispersion extraction.	[29]
$NaHCO_3$	NaH_2PO_4	CNT /polystyrene-divinylbenzene composite	Biosamples	1 mL (for liquid samples) and 6 mL (for reconstituted solid samples)	Alkaloids and flavonoids	The extractant is prepared as effervescent powder (sodium bicarbonate) inside a pipette tip. The proton donor is added to the aqueous sample before manual withdrawal. The effervescence occurs inside the pipette tip dispersing the sorbent.	[30]
$NaHCO_3$	NaH_2PO_4	IL-coated core-shell SiO_2@Fe_3O_4 MNPs	Plasma	10 mL (diluted)	Betablockers	Synthesized sorbent (IL-SiO_2@Fe_3O_4) added separated to effervescent	[31]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						precursors shows better extraction efficiency than adding the components mixed (non-immobilized IL).	
$NaHCO_3$	NaH_2PO_4	$[3C_6C_{14}P]$ $[BF_4]$	Water	10 mL	Benzoylurea insecticides	A tertiary tablet containing the effervescence precursors and the IL is prepared. After the extraction, the solvent is recovered as a solid in the upper part of the centrifugation tube.	[32]
Na_2CO_3	NaH_2PO_4	Ionic liquid nanofluid	Honey and tea	8 mL (honey is 1:10 *w/v* diluted)	Acaricide	A tertiary tablet containing the effervescence precursors and the IL nanofluid is prepared. The solvent is recovered by centrifugation.	[33]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
Na_2CO_3	NaH_2PO_4	[HMIM] [PF_6]	Food samples	10 mL (pretreated sample)	Selenium	The ionic liquid is added to the tablet that also contains the effervescence precursors and magnetic nanoparticles.	[34]
Na_2CO_3	NaH_2PO_4	[HMIM] [NTf_2]	Water	8 mL	Fungicides	The ionic liquid is added to the tablet that also contains the effervescence precursors and magnetic nanoparticles.	[35]
Na_2CO_3	NaH_2PO_4	[BMIM] [PF_6]	Water and milk	7 mL (pretreated sample)	Polybrominated diphenyl ethers	The ionic liquid is added to the tablet that also contains the effervescence precursors and magnetic nanoparticlesFe_3S_4 are used instead of common Fe_3O_4.	[36]
Na_2CO_3	Tartaric acid	[HMIM] [BF_4]	Urine and serum	7 mL (diluted and pretreated sample)	Endogenous steroids	The ionic liquid is added to the tablet that also contains the effervescence precursors and magnetic nanoparticles	[37]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						After the extraction NH_4PF_6, is added to make the IL recovery easier.	
Na_2CO_3	NaH_2PO_4	$[BMIM]_2$ $[Br]_2$	Meat	5 mL (pretreated sample)	Polycyclic Aromatic Hydrocarbons	The tablet contains the effervescence precursors, the IL, the metathesis reagent, and the magnetic nanoparticles $NiFe_2O_4$ nanoparticles are used.	[38]
Na_2CO_3	HCl	[HMIM] [PF_6]	Milk	8 mL (pretreated sample)	Pyrethroids	The ionic liquid is added to the tablet that also contains the CO_2 source and magnetic nanoparticles. HCl is added previously to the sample. The magnetic nanoparticles simplify the IL recovery as it coats the surface of the nanomaterial.	[39]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
$NaHCO_3$	[BMiM][HSO_4]		Tea beverage	5 mL	Triazine herbicides	The IL acts as solvent and H+ donor. After the extraction, NH_4PF_6 is added to make the IL recovery easier. The IL is recovered by centrifugation.	[40]
Na_2CO_3	NaH_2PO_4	[BMIM][$FeCl_4$],	Vegetables	10 mL (pretreated sample)	Arsenite and arsenate	The tablet contains the effervescence precursor and the magnetic IL.	[41]
$NaHCO_3$	Oxalic acid	Sodium nonate	Water	1 L	Steroids	The tablet contains the effervescence precursor and the solvent. Two tablets are added to the sample	[42]
Na_2CO_3	Sulfuric acid	Fatty acid	Several samples	10 mL (pretreated sample)	Antibiotics	The effervescence precursors are added as solutions The fatty acid is recovered by the solidification of	[43]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						floating drop technique.	
Na_2CO_3	Sulfuric acid	Fatty acid	Food samples	6 mL (pretreated sample)	Azo dyes	The effervescence precursors are added as solutions.	[44]
$NaHCO_3$	Citric acid	Sodium octanoate	Beverage	5 mL	Endocrine disrupting chemicals	The tablet contains the effervescence precursor and the solvent. The fatty acid is recovered by the solidification of floating drop technique.	[45]
$NaHCO_3$	Citric acid	Sodium hexanoate	Water	5 mL	Triazine herbicides	Magnetic nanoparticles are added to the tablet to aid the recovery of the sample after the extraction.	[46]
$NaHCO_3$	Acetic acid	DES containing Aliquot 336 and decanoic acid	Food	8 mL (pretreated sample)	Synthetic dyes	DES is dissolved in acetic acid and injected into the sample containing $NaHCO_3$.	[47]
Na_2CO_3	NaH_2PO_4	DES containing choline chloride and phenol	Water	25 mL	Copper	An effervescent tablet is place in	[48]

Effervescent agents		Extractant	Sample		Analytes	Notes	Ref
CO_2 donor	H donor		Type	Amount			
						the extraction vessel. Later, the sample and the DES are introduced into the vessel.	
$NaHCO_3$	Citric acid	DES containing hexyltrimethylammonium bromide and 1-dodecanol	Water	5 mL	EDC	Fe_3O_4 coated with activated carbon nanoparticles is added to recover the solvent after the extraction.	[49]
$NaHCO_3$	Citric acid	DES containing thymol with octanoic acid	Liquid samples	5 mL	Fungicides	The DES is recovered by solidification of DES.	[50]
Na_2CO_3	Formic acid	DES containing formic acid and menthol	Liver	10 mM (pretreated sample)	Ketoprofen, diclofenac	The HBD acts as proton donor in the effervescent reaction while the HBA acts as the solvent.	[51]

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