An Overview: Analysis of ultrasonic-assisted extraction’s parameters and its process

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Abstract. Previously, there were many extraction methods that had been done on extracting the oil such as centrifugation, chilling and thawing, hot and cold extraction, on seeds, waste products and plants. Based on those oil extraction methods (OEM), further study is needed to develop more effective processes as most of the methods were having many disadvantages in term of time, cost, quality and safety. Ultrasonic-assisted extraction (UAE) is used in this project as it is a rapid and effective extraction technique that uses ultrasound to generate rapid movement of solvents, resulting in a higher mass transfer speed as well as acceleration of extraction. Compared to other advanced extraction techniques, UAE is more economic, eco-friendly, and convenient by the reduction of time consumption, higher oil quality and cost reduction. The principle of acoustic cavitation ultrasound which is explained by a series of compression and rarefaction waves induced in the molecules of the medium and the collapse of the bubble during the process of UAE may be discussed. Parameters involved also play the important role in the extraction such as extraction time (ET), extraction temperature (ETem), solvent to material ratio (S/M Ratio) and type of solvent selected. Thus, the paper discusses the application of UAE on oil extraction.

Keyword: Ultrasonic-assisted extraction (UAE), extraction time (ET), extraction temperature (ETem), solvent to material ratio (S/M Ratio).

Introduction of Ultrasonic-assisted Extraction.

Ultrasonic-assisted Extraction (UAE) is a rapid and effective extraction technique that uses ultrasound to generate rapid movement of solvents, resulting in a higher mass transfer speed as well as acceleration of extraction. Compared to other advanced extraction techniques, UAE is more economic, eco-friendly, and convenient (Teng et al., 2016). The statements were supported by Boateng & Lee, 2013 where it is report that UAE is a simple and fast technique, which consumes less energy, time and materials, thus producing more pure products at higher yields (Boateng & Lee, 2013). UAE can be performed under low operation temperature where it can prevent thermal damage to the extracts and preserves the bioactive compounds’ properties in term of its structure and molecule, thus UAE is seen as an ideal option for the edible oil industry (Tian et al., 2013). UAE also no prevent the thermal damage on bioactive compounds but it also avoid the damage in plant materials (Yang et al.,
2017). UAE can be applied in two different techniques, either by using an ultrasonic bath or an ultrasonic horn transducer. Ultrasonic bath which was equipped with a temperature-controlled device which helped the recovery of thermo-sensitive compounds including essential oils (M. Fuad & M. Don, 2016).

There are two ways of ultrasonic irradiation which are direct or indirect contact with sample. Indirect contact means the contact happens through the walls of the sample such as ultrasonic bath system. Direct contact system is more effective in extraction process as it can power up to 100 times better than indirect contact or ultrasonic bath (Medina-torres et al., 2017).

Recently, there have been many reports on the application of UAE in extraction of trace organic compounds from soil, animal and plant tissues (Teng et al., 2016). Besides, UAE is also well recognized to exhibit great effects on the rate of various extraction processes in food, pharmaceutical and cosmetic industries (Yang et al., 2017).

The advantageous of using UAE are it is easy to handle, safe in term of atmospheric pressure and ambient temperature, moderate use of solvent and it also reproducible (Medina-torres et al., 2017). It could also be operated at moderate temperature which is suitable for heat-sensitive compounds (Ilghami et al., 2015). In contrast, the disadvantages of UAE are it requires filtration step and possible degradation of compounds at high frequencies.

**Ultrasonic-assisted Extraction Process.**

UAE is based on the principle of acoustic cavitation ultrasound that induced via a series of compression and rarefaction waves in the molecules of the medium through which it is capable of damaging the cell walls of plant matrix as well as favouring the release of bioactive compounds (Medina-torres et al., 2017; Vinatoru et al., 2017). During sonication, acoustic cavitation produces cavitation bubbles which causes the breaking of the plant cell wall and eventually allows easy percolation of solvent into the extractable material (Boateng & Lee, 2013). In details, the use of ultrasound or sonication is to break the cell membranes that has the advantage of reducing considerably the extraction time and increasing the yield extracted. The application of ultrasound ruptures the cell wall structure and increases the diffusion through membranes; thus, the cell lyses and hence facilitates the release of cell contents (Falleh et al., 2012). The cavitation bubbles will form at sufficient high power which the rarefaction cycle may exceed the attractive forces of the molecules of the liquid. Such bubbles grow under a process known as rectified diffusion i.e. small amounts of vapour (or gas) from the medium enters the bubble during its expansion phase and during compression, it is not fully expelled. It is the fate of these bubbles when they collapse in succeeding compression cycles which generates energy. The symmetric bubbles are created in a homogenous liquid and their collapse in the bulk liquid are also symmetric leading to localized hot spots (~5000 K and ~2000 atm) and another one is an asymmetric bubbles which cavitation in a heterogeneous extraction mixture is that the collapse of the bubbles is asymmetric resulting extremely high speed jets of solvent targeting the vegetal material and this what makes UAE so effective. As soon as a bubble
collapses near a surface where this could be vessel wall, herbal particles or any suspended material in the liquid, that bubble deforms taking a doughnut- shape i.e. the collapse is asymmetrical and forming a high velocity jet, impacting the wall with the potential to sweep particles away from the surface or indeed cause actual damage. This is one of the main reasons why ultrasound is very effective for surface cleaning. During the sonication of a vegetal material in a solvent, the suspended solids promote asymmetric bubble collapse which generates jets of solvent towards herbal particles which boost more efficient extraction from them. Asymmetric bubble collapse is the key parameter of ultrasonically assisted extraction and as in many ultrasonic processes the acoustic power applied must be large enough to overcome the cavitation threshold (Mason et al., 2017).

Furthermore, ultrasounds also use a mechanical effect which promotes better penetration of solvent into the sample matrix thus allowing higher diffusion rates across the cell wall (M. Fuad & M. Don, 2016).

Parameter Affecting the Ultrasonic-assisted Extraction

Extraction Time (ET)

During the UAE process, the solvent will be contacted by the solutes or the samples. Then, the efficiency of extraction is influenced by the interaction time of two phases. There are two phases which are “washing” step and “slow extraction”. The first phase, “washing” step is operated for the first 10-20 minutes of extraction where the surface of the matrix that containing the dissolved of soluble component will be carried out and around 90% of extraction can be done at this stage, thus it is rapid extraction rate. The second phase called as a “slow extraction”, where the diffusion process occur within a 60-100 minutes as the mass transfer of the solute from the matrix diffused into the solvent (Medina-torres et al., 2017).

Based on the previous studies, many researchers concluded the extraction time is around 30 minute during UAE process (Ghafoor et al., 2009; Liu et al., 2017; Boateng & Lee, 2013; Samaram et al., 2013; Teng et al., 2016; Tian et al., 2013; Yang et al., 2017). It also reported that some researches obtained the optimal extraction time more than 30 minute (Li et al., 2016; Tang et al., 2010; Zhang et al., 2009; Zhou et al., 2017).

At the peak of the extraction, the extracts is greatest due to the fully complete cracking of the seed cells due to acoustic cavitation effects during the early stage of extraction, which it can cause a better solvent penetration into the cells and facilitating the release of oil within the cells into the exterior solvent (M. Fuad & M. Don, 2016). Other than that, the production yield increased as the extraction time increased because a longer time will give the ultrasound wave more time to disrupt the cell walls and release the cell contents (Li et al., 2016). However, at certain point of extraction time, the extraction yield will be constant or declined as the system is said to be in equilibrium state from this point onwards. As it is under this state, the oil starts to reabsorb towards the residue of the seed which lowers the yield (Buddin et al., 2018). The statement was supported by stating that target components also readсорb into the ruptured tissue particles due to their relatively large specific surface
areas, lowering yields (Tian et al., 2013). Besides, prolonged extraction time may increase the chances of decomposition of samples and also potentially increase the loss of solvent by vaporization, which can directly affect the loss of mass transfer during extraction (Yang et al., 2017). Therefore, longer extraction time may give a negative result and unnecessary results once the maximum extraction yield has been achieved.

**Extraction Temperature (ETem)**

Temperature is one of the main factors that involved in the UAE process. There were also many researches that had been done by using this parameter as a factor to produce greater yield of extraction. Most of the researches reported that optimum temperature is between 30℃ to 50℃ to get a higher yield (Buddin et al., 2018; M. Fuad & M. Don, 2016; Tian et al., 2013; Yang et al., 2017). The temperature depends on the materials used in extraction.

The yield extraction increased when the extraction temperature was increased. Higher ultrasonication temperature could lead to higher diffusion coefficient of the desired compounds and improve compounds’ solubility in the solvent (Zhou et al., 2017). There are several factors that improve the yield oil which are the matrix bonds ruptures induction, increase of the compound solubility, rate of solvent diffusion, mass transfer and reduction in viscosity and tension of the solvent (Medina-torres et al., 2017). Other than that, higher temperatures would elevate the speed of bubble collapse in the solvent, and this would promote solvent penetration into the cell tissue and speed up the release of the cell contents into the extraction solution (Li et al., 2016).

In contrast, further increment of the temperature causes the reduction of the yield due to cushioning effect meaning that the vapour pressure at elevated temperatures facilitated in the formation of vapour- filled bubbles and as a result, the implosion of those bubbles was cushioned. The cavitation effect is said to be less efficient at high extraction temperature, hence has significantly lowered the yield (Buddin et al., 2018). Besides, as temperature increased, the solvent viscosity and density decreased resulting in an acceleration of mass transfer and the number of cavitation bubbles within the fluid increased forming a cohesive force reducing the tensile strength of the liquid as a result of decreased solvent viscosity (Tian et al., 2013). Moreover, excessively high temperature could sometimes degrade bioactive compounds in the extracts, which decreases the yield of antioxidant (Zhou et al., 2017). Thus, this cushioning effect would eventually cause the ultrasound cavitation effect to be less efficient (M. Fuad & M. Don, 2016).

**Ultrasonic power of Extraction**

The yield of extraction in UAE process was also influenced by ultrasonication power. As increasing the ultrasonication power of UAE will increase the yield of extraction. Sonication is widely used for extraction of various compounds from plant material and generates microscopic bubbles. The amplitude of ultrasonic waves traveling through the solvent are positively correlated with the type and number of bubbles created (Tian et al., 2013). Then, the larger amplitude ultrasonic wave traveling through extraction solvent will create more bubbles and more collapse as well as violent shock wave
and high-speed jet might be generated to disrupt the cell walls where exhibiting the increment of oil yield and it is attributed to the formation of cavitation bubbles and violent explosion of those bubbles onto the cell surface. Therefore, the penetration of extraction solvent into cells became more efficient, resulting in more release of target components from cells into the mass medium (Tian et al., 2013; Yang et al., 2017).

However, extraction efficiency tended to decrease when ultrasonic power was higher than the optimal ultrasonic power. The degradation or decomposition of the antioxidant ingredients in the extracts happens due to the excessively of high ultrasonic power (Zhou et al., 2017). The phenomenon happens due to the elevation of the solvent temperature as well as vapor pressure which lead to a declination in the cavitation intensity. This occurrence did not produce a conducive condition for the enhancement of the extraction process and improvement of the oil yield (M. Fuad & M. Don, 2016).

**Liquid to Solid Ratio of Extraction (S/M)**

Liquid to solid ratio (S/M) is one of the important factors that must be carried out to gain the higher yield in the extraction process. During extraction, the liquid–solid ratio of a reaction mixture can affect the degree of interaction between the solid and the solvent, which in turn the best yield can be achieved when the solution reaches saturation concentration (Li et al., 2016). The statement is supported which a particular degree of enhancement of S/M ratio might improve efficiency of extraction, which is possible because of a greater concentration difference (Zhou et al., 2017).

The percentage of oil yield increases as well as the increment of solvent volume in the system. The mass transfer driving force between solid and liquid phase was enhanced as the S/L ratio increased (Buddin et al., 2018). Furthermore, based on the mass transfer principles occurred in UAE, the driving force of mass transfer is considered to be the concentration gradient between the solid and solvent (Yang et al., 2017). In details, the significant increase in the oil yield at a higher L/S ratio resulted from a greater driving force during the mass transfer of oil which caused by a larger concentration gradient between the interior cells and the exterior solvent. This driving force lead to the diffusion thus allowing the oil to dissolve in the solvent at a higher rate (M. Fuad & M. Don, 2016).

However, the oil yield did not significantly increase as a larger amount of solvent will not change the driving force due to an excessively increase in the ratio (Tian et al., 2013). Besides, high amounts of solvents means an increasing the cost for subsequent operations, such as the concentration and filtration of the extracts obtained, as well as an increase in the amount of waste generated (Medina-torres et al., 2017).

**Type of Solvent**

Solvent type plays an important role in extraction of oil essential. Solvent selection plays a crucial role in UAE, mainly due to its significant effect on cavitation effectiveness, as well effectiveness, as well as in acoustic energy transference to reactants (Giacometti et al., 2018). The initiation of cavitation in a liquid requires the negative pressure during the rarefaction cycle to counter the cohesive
forces between molecules composing the liquid. Increasing of these molecular interactions is induced by the rise of viscosity where it is raising significantly the cavitation threshold. In this condition, samples will have high amplitude due to the high viscosity because the resistance of the sample to the movement of the ultrasonic device also increase as increasing at the level of samples’ viscosity. A low vapor pressure of solvent is preferable in UAE as the collapse of cavitation bubble will be more aggressive. However, vapor pressure depends on the temperature of the liquid medium (Chemat et al., 2017). In addition, the yield of oil extraction will drop as the polarity of the solvent increase thus the lowest polarity will give a positive results of the yield (M. Fuad & M. Don, 2016).

Furthermore, there were some researches that had been done according the solvent type in the application of UAE in extraction of oil. Based on the previous studies, the researches were done using n-hexane (M. Fuad & M. Don, 2016; Yousuf et al., 2018), methanol (Altemimi et al., 2016; Gimbut et al., n.d.; Jakobe et al., 2015; Kim et al., 2019) and the most solvent used was ethanol (Cassia et al., 2018; Charpe & Rathod, 2014; Gimbut et al., n.d.; Hadiyanto, 2016; Jing et al., 2015; Kim et al., 2019; Prommajak et al., 2014; Tuncay, 2015; Yang et al., 2017; Zhou et al., 2017).

Ethanol is used widely in the oil extraction industry because it is the safest solvent for the environment and people, and is widely employed in the food industry (Zhou et al., 2017).

**Conclusion**

Ultrasonic-assisted Extraction (UAE) is a rapid and effective extraction technique as it is simple and fast technique, which consumes less energy, time and materials, thus producing more pure products at higher yields. Besides, the process of UAE is based on the principle of acoustic cavitation ultrasound which is propagated via a series of compression and rarefaction waves induced in the molecules of the medium. Several parameters should be taken as yielding the highest extracted oil such as 30 minutes (extraction time), 30°C - 50°C (temperature) and ethanol as the solvent (type of solvent). More advantages on UAE as it easy to handle, safe in terms of atmospheric pressure and ambient temperature, moderate use of solvent and reproducible.

| Parameters               | Optimum Value of Extraction |
|--------------------------|-----------------------------|
| Extraction Time (min)    | 30                          |
| Extraction Temperature (°C) | 30-50                      |
| Type of Solvent          | Ethanol                     |

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