Journal of Applied Sciences

ISSN 1812-5654

science alert

ANSInet
an open access publisher
http://ansinet.com

Journal of Applied Sciences 11 (13): 2444-2447, 2011
ISSN 1812-5654 / DOI: 10.3923/jas.2011.2444.2447

Solid Liquid Extraction of Jatropha Seeds by Microwave Pretreatment and Ultrasound Assisted Methods

Sepidar Sayyar, Zurina Zainal Abidin, Robiah Yunus and Azhari Muhammad
Department of Chemical and Environmental Engineering, Faculty of Engineering,
UPM 43400 Serdang, Selangor, Malaysia

Abstract: *Jatropha curcas* has a variety of uses which are of great economic significance. Jatropha oil can be used as fuel alternative and for making biodiesel that is supposed to overcome the source limitation problem. In this study, conventional, ultrasound assisted and microwave pretreatment solid liquid extraction of Jatropha seed were studied in terms of amount and quality of the extracted oil. The free fatty acid content which is an important oil quality index was also investigated for the obtained oil. Both ultrasonication and microwave pretreatment of the seeds had a positive effect on amount of yield. However, by application of ultrasound, more oil could be extracted compared with that obtained by conventional and microwave pretreatment extraction methods. The maximum amount of oil which could be extracted by conventional, ultrasound assisted and microwave pretreatment methods were 47.33, 51.4 and 49.36%, respectively. Regarding the quality, oil extracted by conventional, ultrasound assisted and micmicrowave pretreatment extraction methods did not show any significant difference in terms of Free Fatty Acids (FFA) content.

Key words: Jatropha, solid liquid extraction, ultrasound extraction, microwave pretreatment extraction, biodiesel

INTRODUCTION

Jatropha curcas is a perennial, drought resistant plant which is native to Central America but grows well in tropical and subtropical regions of the world. The oil from the seeds of the plant has been used as an industrial raw material since long time ago. The oil can be used for producing soap, cosmetics and candles. More importantly, Jatropha oil can be used as fuel alternative and for biodiesel production that is supposed to overcome the world energy crisis.

Solid liquid extraction, mostly know as leaching in industry, involves separation of solute component from the solid phase. The solid and liquid phases are retained in intimate contact so that solute diffuses from the solid to the liquid phase. It was already proven that by considering the cost and efficiency, solid liquid extraction is the most common efficient and suitable technique in producing oil for fuel and biodiesel production (Forson *et al*., 2004; Yunus *et al*., 2006).

Among the newer techniques used in extraction technology, ultrasound has shown to be an effective method to assist the extraction of oil and other components from vegetables. Ultrasound is a mechanical wave in a frequency range of 16 kHz to 100 MHz (Cains *et al*., 1998; Mason and Cordemans, 1996) utilized to disrupt plant cell walls and thus facilitate the release of contents and improves mass transport of solvent from the solution into the particles (PORIM, 1995). According to earlier experiments (Vinatoru, 2001; Li *et al*., 2004), it is proven that use of ultrasound in solvent extraction produces a greater yield and safer procedure.

Microwave pretreatment of the oilseeds was found to have a positive influence on amount of the oil yield. In microwave heating method, Microwaves penetrate deeply into the materials, propagate there and get converted to heat; therefore, the waves heat the materials from inside rather that just at the surface. This phenomenon is the major difference between microwave heating and heating by the other methods. Microwave pretreatment is reported as an effective method for increasing the oil yield (Ramesh *et al*., 1995; Uquiche *et al*., 2008). Microwaves rupture the cell membrane of the seed and induce pores in its structure. As a result, penetration of solvent into the cells as well as release of solute from inside the seeds gets facilitated and oil recovery will be improved.

The objective of this study was to investigate the effect of ultrasound and microwave pretreatment on the process of solid liquid extraction of Jatropha seeds and on the quality of the obtained oil.

Corresponding Author: Sepidar Sayyar, Department of Chemical and Environmental Engineering, Faculty of Engineering, UPM 43400 Serdang, Selangor, Malaysia Tel: +60176104420

MATERIALS AND METHODS

Raw material preparation: *Jatropha curcas* L. seeds were cracked and the shells were carefully removed. The kernels were grinded with a sieve plate and shaker grinder (Fritsch Cutting Mill P19) by using 0.5 sieve plates. Particles above the sieve were collected and used as coarse size seeds for extraction.

Conventional solid liquid extraction of jatropha seeds: Twenty gram of grinded meal was extracted with n-hexane. The extraction temperature was set to boiling point of the solvent (around 68°C) while the solvent to solid ratio was fixed at 6:1. The extraction was carried out in eleven extraction cycles of 15 min to 8 h. At the end of each extraction cycle, the micelle was filtered using a vacuum filtration (Millipore glass base and funnel) to eliminate suspended solids. Subsequently, the solvent was evaporated using rotary vacuum evaporator (Laborota 4000) and was collected in the receiving flask. The oil which was remained in the sample flask was weighed after the process was completed. The percentage of extracted oil was calculated by dividing the amount of obtained oil by the amount of the seeds multiply by 100. All experiments were repeated at least twice.

Ultrasound assisted extraction of jatropha seeds: To study the effect of ultrasonication on solid liquid extraction of Jatropha seeds, 20 g of coarse size seeds were extracted with 120 mL hexane at 68°C. The extractor was immersed into an ultrasonic cleaning bath operating at 40 KHz frequency. The extraction time varied between 15 min to 8 h. At the end of each extraction cycle, the amount of oil yield was determined.

Microwave pretreatment extraction of jatropha seeds: The effect of microwave preheating on the extraction yield was studied using the method described by Li *et al.* (2004). A 2450 MHz, 23 L microwave was used for heating the seeds. Jatropha seeds were spread out in form of a ring on the glass turn table of the microwave and were heated for periods ranging from 2 to 6 min. After microwave treatment, 20 g of the irradiated seeds were extracted by conventional method for 8 h.

Free fatty acids determination: Free Fatty Acid (FFA) is one of the most frequently determined quality indices during production, storage and marketing of vegetable oils which are used for fuel and biodiesel production (Williams and Macgee, 1983). The percentage of free fatty acid in most types of fats and oils is expressed as oleic acid (PORIM, 1995). The percentage of free fatty acids content of the extracted oil was determined using PORIM test methods (PORIM, 1995). The samples were dissolved in hot isopropyl alcohol and titrated with sodium hydroxide solution using phenolphthalein as indicator. The percentage of free fatty acids is calculated based on the following formula:

$$\text{FFA}(\%) = \frac{V \times N \times 28.2}{\text{Mass (gr of sample)}} \quad (1)$$

where, V is Ml of NaOH required, N is normality of the sodium hydroxide solution and 28.2 corresponds to the equivalent weigh of free fatty acids in which results are to be expressed (here, oleic acid).

RESULTS AND DISCUSSION

Comparison of conventional and ultrasound assisted extraction methods: Figure 1 shows variation of the oil yield with time for conventional and ultrasound assisted solid liquid extraction of Jatropha seed. For both methods, the rate of extraction was very fast at the beginning of the operation, followed by a slow increasing rate for the remaining period of extraction period.

In conventional extraction, around 40% of extractable oil is obtained in the first hour and most of the oil is extracted after 6 h. However, the maximum amount of oil which could be obtained by this method was 47.33% which was achieved after 8 h of extraction.

In comparison to conventional method, the amount of oil yield was higher and the rate of extraction was more rapid with ultrasound at all time intervals. Ultrasound assisted extraction gives a larger yield at the early stage of extraction compared with conventional extraction. The amount of oil that was extracted by ultrasound assisted method in first 15 min of extraction is around 2 times more than the yield obtained by conventional method. 32.5 and 18.55% oil was obtained after 15 min extraction by

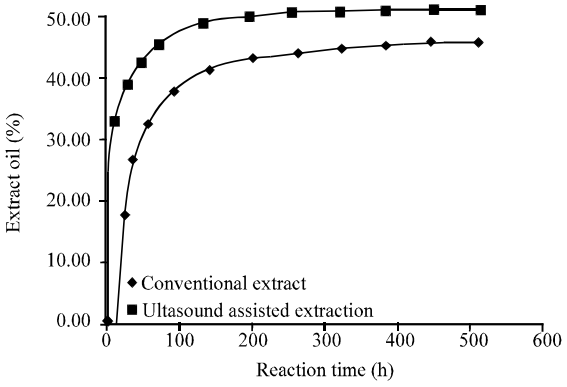

Fig. 1: Comparison of ultrasound assisted with conventional solid liquid extraction of Jatropha seed

ultrasound assisted and conventional extraction method respectively. Furthermore, the amount of extracted oil by ultrasonication for 2 h is more than that by conventional extraction in 8 h. 48.5% oil can be obtained after 2 h of ultrasound assisted extraction against 47.33% which is the highest yield that can be obtained after 8 h of conventional extraction. Moreover, application of ultrasound has shortened the reaction time as maximum yield of 51.4% was reached after 5 h of ultrasonication.

Generally, when the meal is exposed to the fresh solvent, the free oil on the surface of seeds is solubilised and gets extracted quickly inducing a fast increase in the extraction rate. Furthermore, since the oil concentration is low in the solvent at the beginning of the extraction process, the oil diffuses quickly from the seeds to the liquid phase. As the time passing by, the extraction rate becomes slower. Firstly, because of the external diffusion phenomenon which means diffusion should take place through stagnant liquid film around the solid particles. Secondly, due to increase of oil concentration in the solvent, diffusion rate decreases and consequently the extraction gets slower.

The results achieved from ultrasound assisted extraction method indicate a significant reduction of extraction time as well as increase of extraction efficiency. Therefore, application of ultrasound with solid liquid extraction can be an effective and economic method to increase the yield and reduce the process time.

Effect of microwave pretreatment on conventional solid liquid extraction: Effect of microwave pretreatment of Jatropha seeds for 2 to 6 min before extraction on amount of yield is shown in Fig. 2. Irradiation of seeds for 2 min before extraction did not have any positive effect on yield.

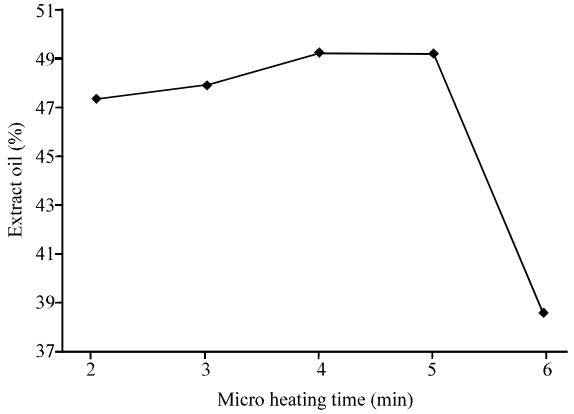

Fig. 2: The effect of microwave pretreatment time on amount of extracted oil using hexane as solvent, 6:1 ratio and coarse size seeds at boiling point temperature for 8 h of extraction

The amount of oil yield after 8 h of extraction was 47.33% which is same with that obtained from unirradiated seeds. By increasing the treatment time to 3 min, the amount of yield increased slightly to 48%. Increase in the duration of irradiation to 4 min enhanced the oil recovery to 49.36%, around 2% more compared with that of untreated seeds.

Preheating the seeds for 6 min before extraction decreased the amount of yield dramatically to 38.6%. Some proportion of the seeds got burnt and roasted after 6 min irradiation that was presumably resulted in less penetration of solvent into the sample matrix and low oil recovery.

Based on the results, microwave pretreatment of Jatropha seeds before solid liquid extraction can improve the oil recovery up to 2%. Seed pretreatment for 4 min enabled 49.3% oil to be extracted.

Free fatty acids percentage: Table 1 shows the free fatty acid percentage of Jatropha oil extracted immediately and one week after grinding through conventional, ultrasound assisted and microwave pretreatment methods.

There is not any significant difference between the free fatty acid content of the oils which were extracted by the three methods. The free fatty acid percentage of the oils extracted by conventional, ultrasound assisted and microwave pretreatment techniques was around 0.6 to 0.7% when extraction was carried out immediately after grinding the seeds and around 7% when the grinded seeds were extracted after one week.

The results indicating that ultrasound assisted extraction and microwave preheating of the seeds do not have any negative effect on the quality of the oil in terms of free fatty acid content. However, storing the seeds after grinding resulted in a reduction in the quality of the extracted oil. Free fatty acid content of the oil has increased considerably by more than 6% when the seeds were extracted one week after grinding. It might be because of enzymatic degradation, oxidation and hydrolysis occurred during the storage period.

Using vegetable oils with high free fatty acids and viscosity as a direct fuel may cause fatal problems for the engine. Therefore, the lower is the free fatty acid content; the more appropriate is the oil for being used as fuel alternative. Furthermore, in biodiesel production, free fatty acid content of the oil should be less than one percent to

Table 1: FFA content of the oil obtained by conventional, ultrasound assisted and microwave pretreatment extraction methods

	Free fatty acid (%)		
Storage before extraction	Conventional	Ultrasound	Microwave
Extracted immediately	0.62	0.67	0.63
Extracted after one week	6.86	6.81	6.97

reach satisfactory transesterification reaction using alkaline catalyst (Tiwari *et al*., 2007). A pretreatment process is required if the oil contains significant amounts of free fatty acid since free fatty acids form soap with alkaline catalyst and prevents separation of the biodiesel from the glycerin fraction.

According to the free fatty acid test data, the free fatty acid content of the *Jatropha curcas* L. oil is quite low, so it can be used as fuel alternative specially for biodiesel production provided that the seeds get extracted immediately or at least within a short time after grinding. Pretreatment process which is requisite for high free fatty acid content oils in biodiesel production, can be eliminated by extracting the seeds after short storage duration.

CONCLUSION

In this study, the effect of microwave pretreatment and ultrasound on solid liquid extraction of Jatropha seed was studied. Application of ultrasound clearly promoted the release of oil and enhanced the yield of extraction. Furthermore, ultrasound significantly shortens the extraction time. Preheating the seeds for 4 min by microwave before the extraction improved the oil recovery up to 2%. Ultrasonication and microwave did not affect the oil quality in terms of free fatty acid content. Storing the grinded seeds for one week before extraction left a remarkable negative effect on quality of the produced oil while the quality of the oil was much better by extracting the seeds immediately after grinding.

REFERENCES

Cains, P.W., P.D. Martin and C.J. Price, 1998. The use of ultrasound in industrial chemical synthesis and crystallization. 1. Applications to synthetic chemistry. Org. Proc. Res. Dev., 2: 34-48.

Forson, F.K., E.K. Oduro and E. Hammond-Donkoh, 2004. Performance of jatropha oil blends in a diesel engine. Renewable Energy, 29: 1135-1145.

Li, H., L.O. Pordesimo, J. Weiss and L.R. Wilhelm, 2004. Microwave and ultrasound assisted extraction of soybean oil. Trans. ASAE, 47: 1187-1194.

Mason, T.J. and E.D. Cordemans, 1996. Ultrasonic intensification of chemical processing and related operations: A review. Trans. I Chem. E, 74: 511-516.

PORIM, 1995. PORIM Test Methods. Palm Oil Research Institute, Malaysia, pp: 25.

Ramesh, M., P.H. Rao and C.S. Ramadoss, 1995. Microwave treatment of groundnut (*Arachis hypogaea*): Extractability and quality of oil and its relation to lipase and lipoxygenase activity. LWT Food Sci. Technol., 28: 96-99.

Tiwari, A.K., A. Kumar and H. Raheman, 2007. Biodiesel production from jatropha oil (*Jatropha curcas*) with high free fatty acids: An optimized process. Biomass Bioenergy, 31: 569-575.

Uquiche, E., M. Jerez and J. Ortiz, 2008. Effect of pretreatment with microwaves on mechanical extraction yield and quality of vegetable oil from Chilean hazelnuts (*Gevuina avellana* Mol.). Innovative Food Sci. Emerg. Technol., 9: 495-500.

Vinatoru, M., 2001. An overview of the ultrasonically assisted extraction of bioactive principles from herbs. Ultrasonics Sonochem., 8: 303-313.

Williams, M.G. and J. Macgee, 1983. Rapid determination of free fatty acids in vegetable oils by gas liquid chromatography. J. Am. Oil Chem. Soc., 60: 1507-1509.

Yunus, R., P.Y. Chang and L.H. Lee, 2006. Preliminary study on extraction and synthesis of biodiesel from *Jatropha curcas* seeds and soybean. Proceedings of the 1st International Conference on Natural Resources Engineering and Technology, July 24-25, Putrajaya, Malaysia, pp: 671-678.