# A RAPID AND EFFICIENT EXTRACTION METHOD FOR *FOENICULUM VULGARE* MILL.

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**Abstract**

Compounds of medicinal plants have shown tremendous potential to treat various diseases. There are various techniques being used at industrial level for extraction of these compounds to develop the herbal medicines. Microwave assisted extraction (MAE) is a novel technique which has greatly reduced the time for extraction and increased the yield of natural products of plant origins. In presented research work, various parameters such as timing (min) and power (W) of microwaves along with solvent type were considered for extraction of compounds from *Foeniculum vulgare* Mill. Results showed that by increasing the power level (300W, 600W, 900W) of microwaves and exposure timing (1 min, 3 min and 5 min) affect the extraction process to get the maximum yield of 260 mg per gram of powder at 600W after 5min obtained from dichloromethane with less solvent consumption.

**Keywords**: *Foeniculum vulgare* Mill, Microwave Assisted Extraction, Optimization, Time of extraction

## Introduction

Plants are being used to treat various diseases since ancient times. The extraction and characterization of phytochemicals gave rise highly potential drugs. Extraction is the very basic and crucial step for the isolation and purification of chemical constituents of plants (Huie, 2002). There are number of methods commonly used for isolation of different chemicals constituents of plants such as maceration, decoction, infusion, tincture, Soxhlet's extraction.

Selection of extraction method is dependent on type and properties of compounds, therefore to isolate and purify the compounds, selection of the best method for isolation of maximum amount of compounds should be cost effective without any damage to external environment (Mueen, 2008). Demand of phytochemicals is increasing day by day especially in medical field so, there is a need to develop some techniques at advance level such as Microwave Assisted Extraction (MAE). These techniques require shorter time with lesser amount of solvent for maximum amount of targeted compounds (Eskilsson and Björklund, 2000).

Microwave assisted extraction (MAE) is a technique developed in the late 1980s. It has greatly reduced the time for extraction and increased the yield of natural products of plant origins (Mandal *et al.*,

2008). Microwaves are combination of electric and magnetic waves which oscillates in perpendicular direction to each other and frequency range is 0.3-300 GHz. These electromagnetic waves have ability to penetrate into certain materials particularly having moisture inside them or polar components. These can interact with components which show polarity to produce the heat. The heat energy produced during this process has some effects on the molecules by phenomenon of ionic conduction and dipole rotation. Therefore only selective materials are heated based on their dissipation factor or loss of energy and dielectric constant (Chan *et al.*, 2011; Routray and Orsat, 2012).

There are some factors which affect the extraction process of microwave assisted extractor, such as type of solvent, solvent composition, power level, temperature and time of extraction. Temperature and power levels are the most important parameters because each solvent has its own boiling point and solvents are heated in microwave extractor during extraction. These directly affect the efficiency of microwaves (Eskilsson and Björklund, 2000; Nemes, 2012).

*Foeniculum vulgare* Mill. is a herbaceous plant which belongs to the family Apiaceae. Its common name is sweet fennel. It grows at open sunny places (Díaz-Maroto *et al.*, 2006). The plant is widely cultivated in the tropical and temperate regions of the world. It is also grown as a cold-weather crop as it is reasonably frost resistant, and does not develop well in the south of India because of hot climatic conditions (Purwaningsih and Brink, 1999).

Fennel seeds have essential oil in rich amount. Due to its strong flavor and role in pharmaceuticals this is extracted from seeds often and used for medicinal purposes like as antifungal, aphrodisiac, anti-inflammatory, digestive, carminative, stimulant and antispasmodic. It is also used in aromatherapy to relief the patients from headache or muscle pains. It also helps to fresh the mind and recovers the fatigue. It has soothing effect when applied on body to reduce discomfort in digestive system, muscular spasms, comforts the rheumatism and pain of arthritis and detoxify the harmful bodies. Oil vapors relief wind and cramps and comforts the glandular system too (Lawless, 1995).

The presented research work was done to optimize the various factors (timing and efficiency of microwaves along with the solvent used) affecting the extraction of compounds from *F. vulgare* to establish a cost-effective protocol for lesser consumption of time and energy.

**Materials and Methods**

The presented research work was done in Biochemistry Lab of Botany Department in Lahore College for Women University (LCWU). The experimental

work of the research was done in four steps given below.

**Preparation of plant material**

Taxonomically identified seeds of *Foeniculum vulgare* Mill. were taken from National Agricultural Research Center (NARC), Islamabad. Seeds were crushed with help of grinder to form fine powdered plant material. Then the plant material was stored in air tight jars for further experimental work such as extraction of targeted compound and quantification of extracted compound.

**Extraction of compounds by microwave extractor**

In this step, extraction of compounds was carried on Microwave Assisted Extractor (Model MDS-6G). Parameters selected for this technique to optimize the yield of compound were solvent type (Methanol, Hexane, Ethyl Acetate and Dichloromethane), power level (300, 600 and 900 W) and time (1, 3 and 5 Min). The solvent was added to main inner vessel and placed in outer protective vessel. Temperature measuring tube was used to cover the vessel. Then, it was loaded to the master frame horizontally.

Plant sample of 1 g with 20 ml solvent was taken in clean inner vessels. Inner vessels were inserted into the outer protective vessels. Vessel were covered carefully and loaded onto the frame of standard digestion. Master vessel was positioned on its specific place. Sample vessels were loaded according to sequence to keep the balance of main frame. Then temperature, pressure and time were set to run the extractor. After that obtained extract was stored in vials properly covered and labeled, in refrigerator at 4ºC.

**Analysis of data**

The data produced in result of experimental work was collected and analyzed. Analysis was done by considering many factors and parameters. The mean values of the data of different treatments were compared by applying one-way ANOVA with the help of a computer software i.e. SPSS. Duncan's New Multiple Range Test of post hoc test was applied at significant level of 5%.

**Results & Discussion**

**Effect of solvent type**

Selection of an excellent solvent is fundamental in extraction process to give high yield. It depends upon the solubility of compounds, interaction between the solvent and plant material and microwave absorbing properties of the solvents (Letellier *et al.*, 1999). Same solvents can be used to extract compounds with MAE which are used in conventional methods. In presented research work different polar and non-polar solvents were used. Table 1 illustrates the effect of solvent type on extraction by MAE at different timings and efficiency of microwaves.

Methanol (Figure 1a) had extracted the maximum amount of extract

followed by ethyl acetate (Figure 1c), dichloromethane and hexane (Figure 1b and 1d) at 300W and 900W power level of microwaves. While, dichloromethane had the maximum amount of extract than other solvents at 600W power level of microwaves which was 260 mg per gram of powder material. Hexane extracted the lesser amount than all other solvents. It showed that different solvents and efficiency of microwaves have effect on extraction process by MAE. Kiss *et al*. (2000) also elaborated the importance of solvent choice for extraction process by using MAE. Talebi *et al*. (2004) reported that the efficacy and efficiency of microwaves depend upon the selected solvent.

Solvents have different dielectric properties which determines the heating and absorption of microwaves. Microwaves shows the better efficiency of MAE with polar solvents therefore methanol heats up faster and absorb more microwaves than hexane which is transparent to microwave and does not absorb microwaves as methanol.

**Effect of timing**

Another parameter which influences the extraction process is also needed to be optimize. Generally, amount of extract is increased by increasing the timings of extraction but there is a risk of degradation for sensitive compounds (Li *et al*., 2004). In this experiment, various timings of microwave irradiation such as 1, 3 and 5 min were considered. There was clearly increase in the amount of extracts by increasing the exposure timing for extraction purpose. Maximum amount was extracted at 5min of irradiation with methanol as Methanol is polar in nature and heat up tremendously (Figure 1a).

Pan *et al*. (2003) extracted the polyphenols and caffeine from leaves of green tea by microwave assisted extractor and found that amount of extract was increased up to 4 min but further increase in time caused the decrease. Time is very crucial parameter to determine and may change even with change in part of plant. It is affected due to dielectric properties of the solvent.

**Effect of efficiency of microwaves**

Efficiency of microwaves in combination with time of exposure influences the yield of compounds at greater extent. In this experiment, various power of microwaves i.e. 300W, 600W and 900W were studied.

Results shown in figure 1a-d. Methanol and ethyl acetate extracted the maximum amounts of compounds at 900W with increasing the time of irradiation. While, dichloromethane and hexane extracted the maximum compounds at 600 W and the amount was reduced with increasing the time of exposure.

Shu *et al*. (2003) also reported the significant importance of microwaves efficiency. Ginsenosides was extracted

from ginseng root by MAE and efficiency of microwaves was increased from 30W to 150W using short time i.e. 1 and 2 minutes. Significant amount of the ginsenosides was extracted within selected range of microwave power in 1 min of exposure. Latha (2007) reported the effect of microwave power i.e. 300W and 450W on extraction of embelin from from *Embelia ribes*. It was extracted higher in amount at 450W for 80 sec. The power must be chosen correctly to avoid excessive temperature, which could lead to solute degradation and overpressure inside the vessel as shown in figure 2 that interaction of power and solvent type plays an important role.

**Conclusion**

It is concluded from the presented work that microwave assisted extraction (MAE) technique is the most promising technique. It protects the heat sensitive compounds as need lesser time for extraction. It consumes lesser amount of both samples and solvents and saves time too. It reduces the consumption of energy and due to this it has become therapeutically valuable as energy crisis is the world's largest issue. Pharmaceutical companies can use this cost effective technique for the production of drugs from medicinal plants.

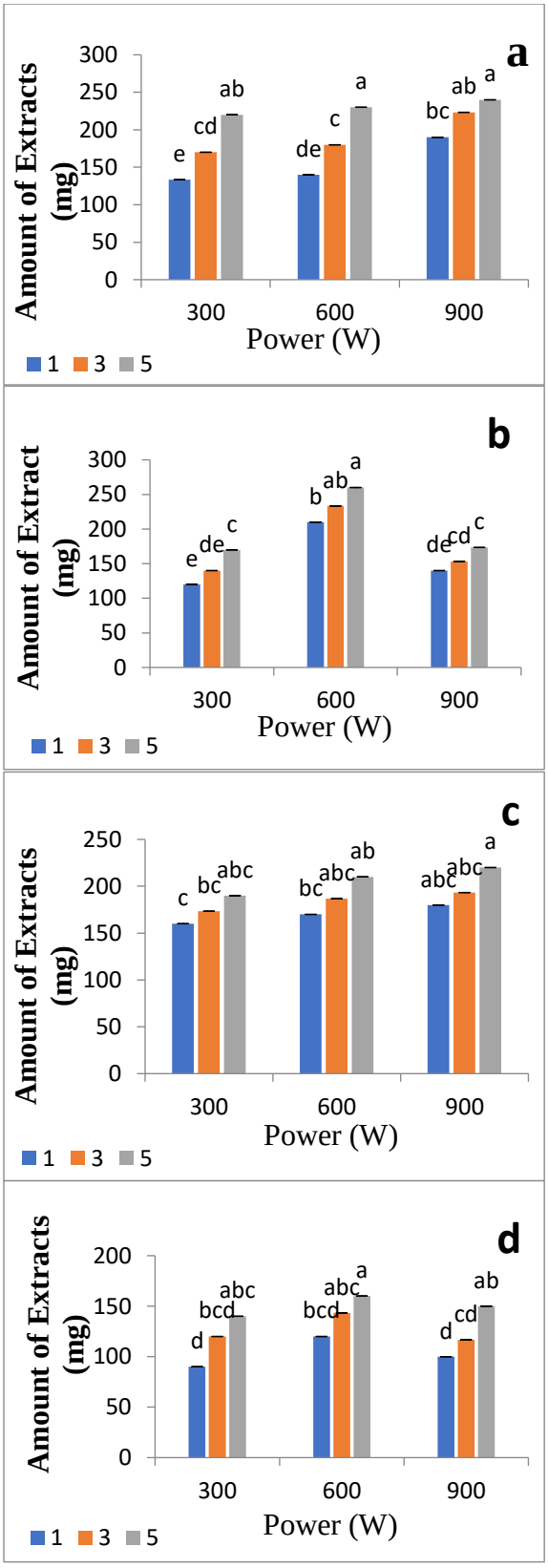


**Figure 1:** Effect of time and power level of MAE on extraction from powder material of *F. vulgare*'s seeds in a) Methanol b) Dichloromethane c) Ethyl acetate d) n hexane

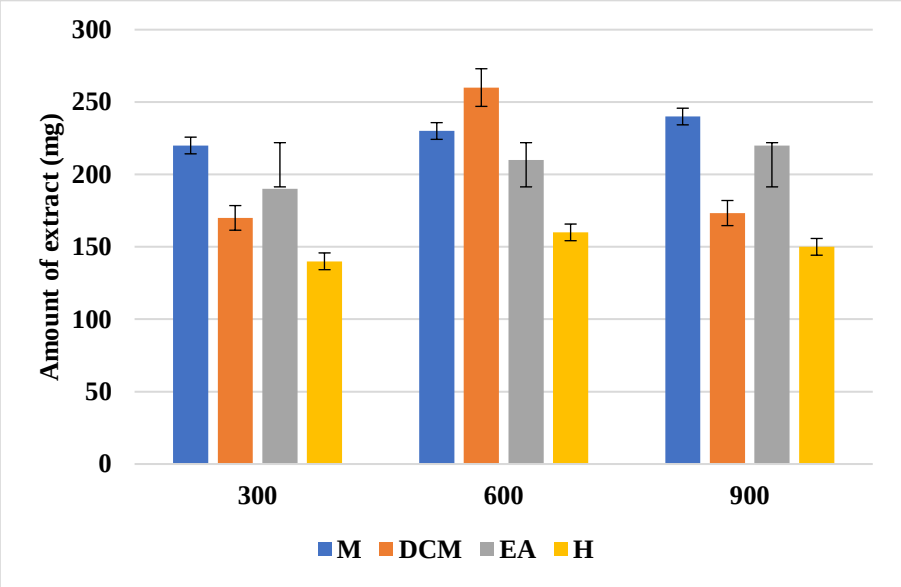


**Figure 2:** Interaction of microwaves and different solvents to get extracts from fennel seeds where M is methanol, DCM is dichloromethane, EA is ethyl acetate and H is n-hexane

## References

Chan, C. H., R. Yusoff, G. C. Ngoh and F. W. L. Kung. 2011. Microwave-assisted extractions of active ingredients from plants. *J. Chromatogr A*, 1218(37): 6213-6225.

Díaz-Maroto, M. C., M. S. Pérez-Coello, J. Esteban and J. Sanz. 2006. Comparison of the volatile composition of wild fennel samples (*Foeniculum vulgare* Mill.) from Central Spain. *J. Agric. Food Chem.*, 54(18): 6814-6818.

Eskilsson, C. S. and E. Björklund. 2000. Analytical-scale microwave-assisted extraction. *J. Chromatogr A, 902*(1): 227-250.

Huie, C. W. 2002. A review of modern sample-preparation techniques for the extraction and analysis of medicinal plants. *Anal. Bioanal. Chem.*, 373(1-2): 23-30.

Kiss, G. A. C, E. Forgacs, T. C. Serati, T. Mota, H. Morais and A. Ramos. 2000. Optimization of the microwave assisted extraction of pigments from paprika (*Capsicum annum* L.) powders. *J. Chromatogr. A*, 889**:** 41-49.

Latha, C. 2007. Microwave-assisted extraction of embelin from *Embelia ribes*. *Biotechnol. Lett.*, *29*(2): 319.

Lawless, J. 1995. The Illustrated Encyclopedia of essential oils the complete guide to the use of oils in aromatic and herbalism. Element Books Ltd., Shaftesbury, Dorset, UK.

Letellier, M., H. Budzinski, L. Charrier, S. Capes and A. M. Dorthe. 1999. Optimization by factorial design of focused microwave assisted extraction of polycyclic aromatic hydrocarbons from marine

sediment. *Fresenius' J. Anal. Chem.*, 364(3): 228-237.

Li, H., B. Chen, Z. Zhang and S. Yao. 2004. Focused microwave-assisted solvent extraction and HPLC determination of effective constituents in *Eucommia ulmodies* (*E. ulmodies*). *Talanta*, 63(3): 659-665.

Mandal, V., Y. Mohan and S. Hemalatha. 2008. Microwave assisted extraction of curcumin by sample–solvent dual heating mechanism using Taguchi L9 orthogonal design. *J. Pharm. Biomed. Anal.*, 46(2): 322-327.

Mueen, A. K. K. 2008. *Text Bookof Industrial Pharmacy*, Chennai, Orient Longman Private Ltd 345.

Nemes, S. 2012. *Practical methods for lignans quantification* (Doctoral dissertation, McGill University Libraries).

Pan, X., G. Niu, and H. Liu. 2003. Microwave-assisted extraction of tea polyphenols and tea caffeine from green tea leaves. *Chem. Eng. Process: Process Intensification*, 42(2): 129-133.

Purwaningsih, H. and M. Brink. 1999. *Foeniculum vulgare* Miller. In: Proseabase [ed. by Guzman, C. C. de \siemonsma, J.]. Bogor, Indonesia: PROSEA (Plant resources of South-East Asia).

Routray, W. and V. Orsat. 2012. Microwave-assisted extraction of flavonoids: a review. *Food and Bioprocess Technology*, 5(2): 409-424.

Shu, Y. Y., M. Y. Ko and Y. S. Chang. 2003. Microwave assisted extraction of ginsenosides from ginseng root. *Microchem. J.*, 74(2): 131-139.

Talebi, M., A. Ghassempour, Z. Talebpour, A. Rassouli and L. Dolatyari. 2004. Optimization of the extraction of paclitaxel from *Taxus baccata* L. by the use of microwave energy. *J. Sep. Sci.*, 27(13): 1130-1136.