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Microwave assisted extraction of Sesamol from *Sesamum indicum* L. (Sesame)

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Abstract

Microwave assisted extraction (MAE) is a rapidly growing extraction technology. It is a process which extracts a solute in a solvent from a solid mixture/micelle/solution substance at optimum conditions of process. The important parameters include solvent type, solvent volume and concentration, extraction time, temperature and microwave power level. In the present study, an antioxidant sesamol was extracted from sesame oil by using MAE process. Optimum conditions for microwave extraction were found at the power level of 800 watt for 7 min of microwave heating with the sample to solvent ratio maintained at 2:25 (w/v). At the optimum processing condition, the amount of sesamol extracted was 374.81 mg/kg of sesame oil. By comparing MAE and the conventional soxhlet extraction processes, it was found that two times more recovery is possible in MAE process within significantly less time than the soxhlet extraction method.

Keywords: Microwave heating, MAE process, HPLC process, sesame, Sesamol

Introduction

Extraction of bioactive compound is performed using either conventional method like soxhlet extraction method or non-conventional methods viz., ultrasound extraction, super critical fluid extraction, enzymatic digestion, pulse electric light extraction, microwave assisted extraction, ohmic heating assisted extraction, extrusion pressurized liquid extraction etc (Shams *et al.* 2020) [14]. Conventional method is used as standard method for newly flourished modus operandi. Common problem for conventional method is prolong extraction time (Ozel and Kaymaz 2004) [13], high labour requirement, resulting in increased production cost. Secondly, it consumes more solvent such (150:1 ml/g, Suja *et al.* 2004) [16] than the non-conventional methods which further enhance the cost of experiment. Conventional approach also consumes more power due to longer process duration. Microwave assisted extraction process eliminates all these drawbacks of conventional method. MAE consumes low volume of solvent as 10:1 (ml/g) (Talebi *et al.* 2004) [18] or 20:1 (ml/g) (Li *et al.* 2004) [10]. This procedure utilizes less time (within few minutes) as compare to soxhlet method. In some cases, MAE took 30 s for optimum recovery (Li. *et al.* 2004) [10]. Time and power factors are interweaved with each other as by elevation of power this process uses less time and vice versa. Aforementioned process is also anti-thermolabile process. It preserves the thermal properties of compound which get lost during long heating of compound in conventional methods (Mandal *et al.* 2007) [12]. By this, we can conclude that MAE process is a substitute of conventional extraction process with advance features.

Sesame seed is highly recognized due to its health benefits like protein source, anti-diabetic, anti-hypertensive property and plays an important role in healthy skin. In India, sesame is widely utilized as sesame oil and sesame sweets. Sesamol is one of the prime lignan (Hemalatha *et al.* 2004) [5] found in roasted sesame seed as well as in roasted sesame oil along with sesamin and sesamol (Lee and Choe 2006) [9]. Sesamol is found only in sesamum plants thus it is a unique characteristic of sesame (Hwang 2005) [7]. During roasting process of sesame seed, sesamol decomposes and forms a new compound called as sesamol and during refining process sesaminol is formed from the sesamol under acidic anhydrous medium of bleaching process (Fukuda *et al.* 1986) [6]. Thus, sesamol is found in roasted sesame seed and oil only, not in raw sesame. Both sesamol and sesaminol have high antioxidant (Fukuda *et al.* 1994) [4] properties which elevate shelf-life of roasted sesame oil. Sesamol is an anti-carcinogenic agent (Kapadia *et al.* 2002) [8]. Natural sesamol is costly than the synthesized sesamol. Quantity of sesamol in roasted sesame seed depends upon quantity of sesamol. In black sesame seed, the ratio of sesamol to sesamin is higher than the white sesame seed.

Black sesame seeds contain lower oil quantity than the white sesame. In Indian varieties, there is significantly low sesamol found (Hwang 2013) [6]. Thus, sesamol is very important bioactive compound detected in roasted sesame seed. Hence, in the present study, MAE was used to extract sesamol from sesame seed and the process parameters were optimized for maximum sesamol recovery.

Materials and Methods

Raw materials and reagents

Black sesame seed was procured from local market of

Kharagpur, West Bengal. Sample was prepared as shown in fig 1. Selection of solvent for MAE process was an important step because solvent must absorb microwave energy as well as it should dissolve sesamol (polar compound) (Li. *et al.* 2004) [10]. So the polar organic solvent, which has high dissipation factor ($\tan\delta$) and low dielectric constant were separated (Table 1). Acetone, Ethanol, Ethyl Acetate, Hexane and Methanol have been taken for trial. All the solvents were purchased from lab solution, Kolkata, West Bengal. 5 g of 98% pure sesamol was purchased from Sigma, India.

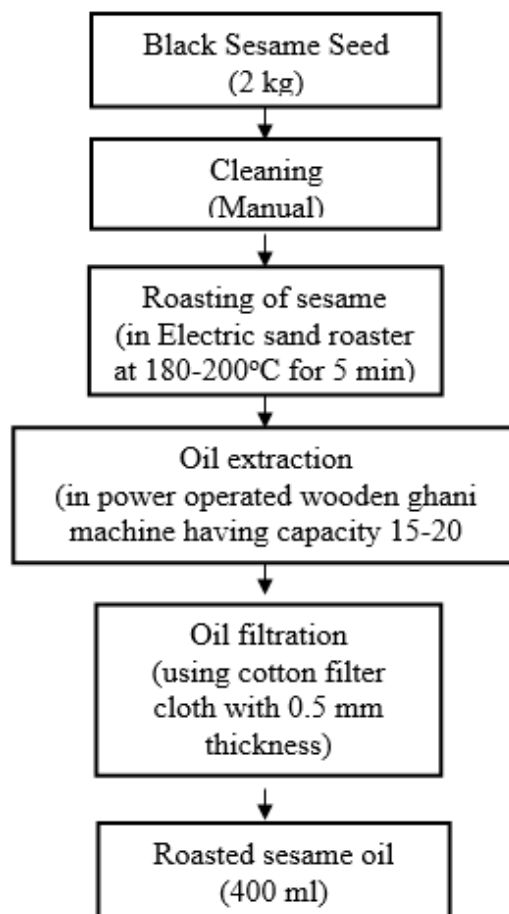


Fig 1: Sample preparation procedure

Table 1: Dissipation factor and Dielectric constant for solvents commonly used in MAE (Source: Mandal *et al.* 2007) [12]

S. No.	Solvent Name	Dissipation factor ($\tan\delta$) $\times 10^{-4}$	Dielectric Constant (ϵ') measured at 20 °C
1	Methanol	6400	32.6
2	Acetone	5555	20.7
3	Ethyl acetate	5316	6.02
4	Ethanol	2500	24.3
5	Hexane	-	1.89

Microwave assisted extraction of sample

MAE apparatus was designed and fabricated in laboratory (Fig. 2) and sample was extracted at different experimental conditions. MAE of sample was executed as per earlier described method by Maran *et al.* (2013). The apparatus was fabricated using convective type domestic microwave oven (Samsung CE74JD model). About 2 g of sample was put into 250ml volumetric flask with varying concentration of solvent. The volumetric flask was placed at center of microwave oven on a glass stand. Condenser was attached to the volumetric

flask to convert the solvent vapour that was produced during microwave heating into liquid phase. Condenser was attached to cooled water source. The sample-solvent mixture was exposed to microwave irradiation at 3 different levels of microwave power as 450, 600 and 800 W. three different levels of time was selected to process the sample as 3, 5 and 7 min. Concentration of solvent was varied as 15, 20 and 25 ml. MAE experiment design was done according to the factorial design method.

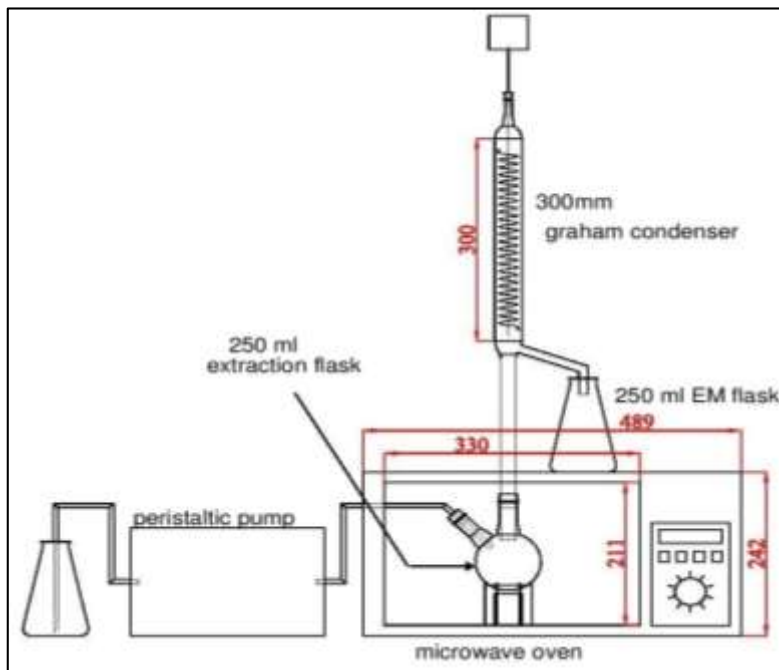


Fig 2: Two-D design of MAE apparatus (all dimensions are in mm)

HPLC process

For analytical measurement of sesamol, HPLC protocol given by Suja *et al.* 2004 [16] was used. HPLC process was performed at Central Research Facility, IIT Kharagpur on an Agilent 1100 series HPLC system with a UV detector and C-18 column thermostated at 35 °C. Samples were injected with mobile phase of 70% of methanol at the flow rate of 1 ml/min. Sesamol was detected at 290 nm wavelength.

Results and Discussion

Selection of solvent

Ethanol and methanol were split after 2 min of heating due to high vapor pressure generation and immiscibility of oil in these solvents. Hexane did not get temperature higher than 60

°C after 7 min of heating at 450 W. It is due to lack of polar compounds in hexane which absorbs the microwave energy. Ethyl acetate and acetone both had showed good properties under microwave heating with sample. The oil sample is more soluble in ethyl acetate due to its emulsion properties so finally ethyl acetate was used as solvent for MAE process.

MAE extraction and effect of various factors on yield

Calibration curve of sesamol for HPLC was obtained using five different concentration of standard solution of sesamol (10, 50, 100, 200 and 500 mg/L) prepared by serial dilution of stock solution of 5 g/L. The straight line was observed with R^2 value as +0.9638 which indicates very good relation of the data fitted to regression line (Fig. 3).

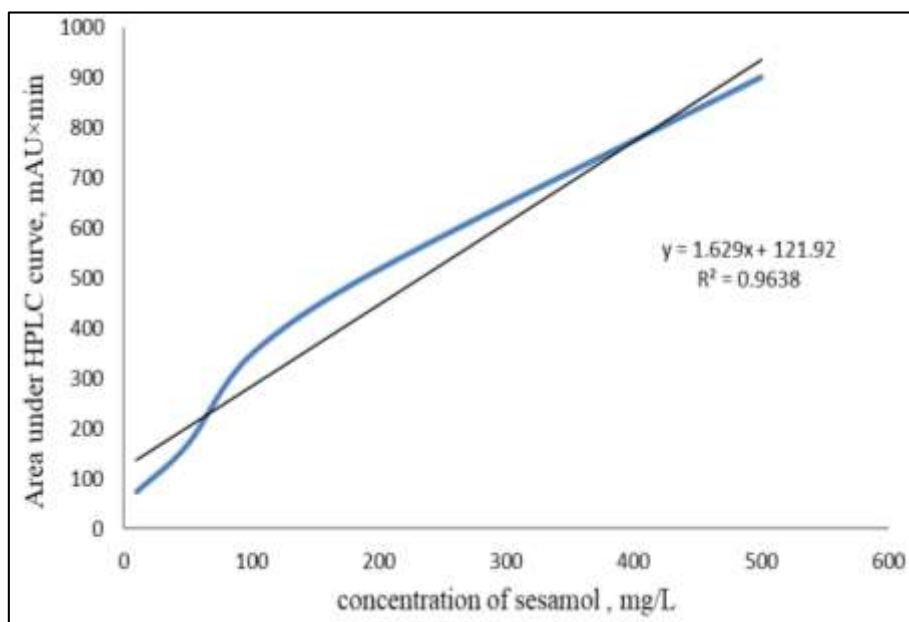


Fig 3: Calibration curve for standard sesamol

Different combinations of power, time and solvent volume were studied to extract the sesamol from the oil samples. The

sample which was heated at 450 watt for 3 min with 15 ml of solvent showed least amount of sesamol (1.99 mg/kg) in it. As

the heating power was increased (800 watt) by keeping the same heating time (3 min) and solvent volume (15 ml), the extracted sesamol amount was increased to 228.00 mg/kg. Similar results were found when solvent volume was maintained at 20 ml or 25 ml while heating power was increased from 450 watt to 800 watt. Enhanced efficiency was also observed at heating time of 5 min or 7 min while increasing the heating power from 450 watt to 800 watt which indicated that heating power affected the extraction efficiency. Fig. 4(a) describes change in extraction yields with change in processing time and solvent volume at 800 W microwave heating.

While examining the heating time factor, it was found that as the heating time was increased from 3 min to 7 min by maintaining heating power at 450 watt and solvent volume of 15 ml, the amount of extracted sesamol was increased from 1.99 mg/kg to 3.55 mg/kg. This rise in the yield was less as compare to heating power factor because at low heating power, sample was not heated up-to the point where maximum amount of sesamol could be leached into the solvent even-after increasing the heating time. When the

similar experiment was done for solvent volume as 20 ml or 25 ml at 450 watt, the increment in Sesamol amount was observed. Similarly, enhanced efficiency was observed when extractions were done at constant heating power of 600 watt or 800 watt while increasing the heating time from 3 min to 7 min. At higher heating power when heating time was increased, yield was more as compare to low heating power which indicated the synergistic effect of the heating power and heating time (Fig 4(b)). Hence, when both the factors (time and power) were increased simultaneously then more yields were obtained at higher amount of solvent.

When the extracted amount for different volume of solvents were compared, it was found that as solvent volume was increased from 15 ml to 25 ml by keeping the heating power at 450 watt and heating time as 3 min, extracted sesamol amount was increased from 1.99 mg/kg to 220.98 mg/kg which clarified that solvent volume affects the extraction efficiency. Similarly, when increased amount of solvent volume were used at 600 watt or 800 watt of heating power, then more extracted sesamol was observed (Fig. 4(c)).

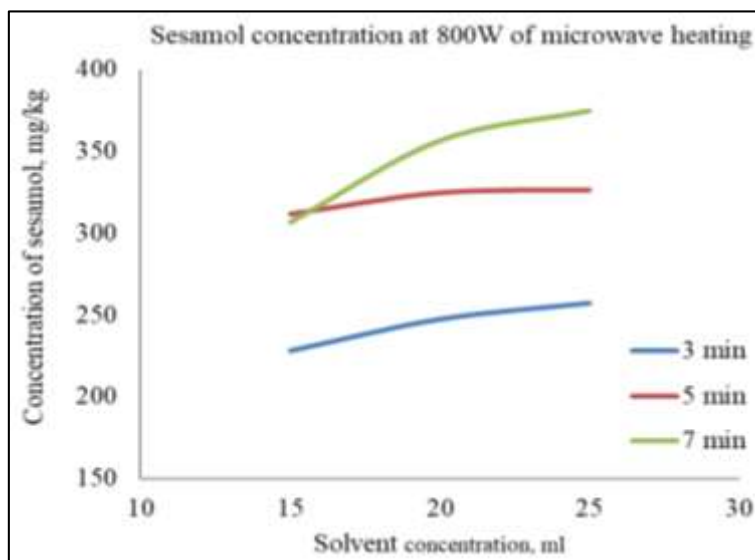


Fig 4(a): Effect of volume of solvent and heating time on concentration of sesamol at constant heating power of 800 W. Sesamol concentration increased as heating time and volume of solvent were increased simultaneously.

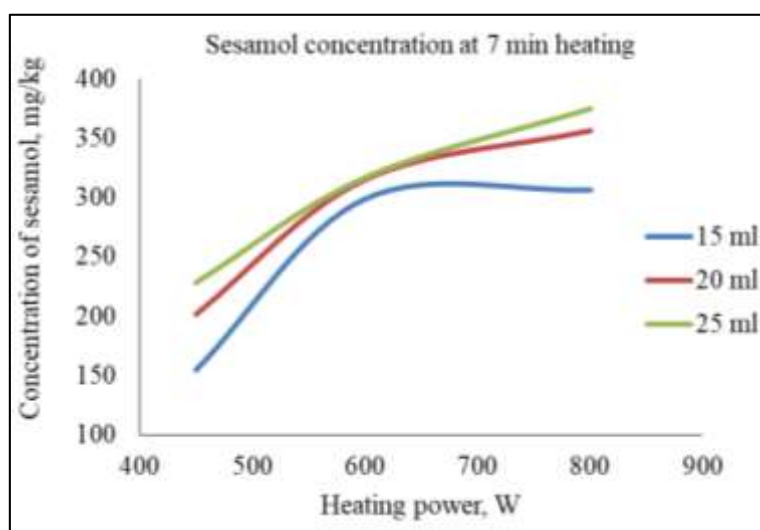


Fig 4(b): Effect of volume of solvent and heating power on concentration of sesamol at constant time. Sesamol concentration increased as heating power and volume of solvent were increased simultaneously.

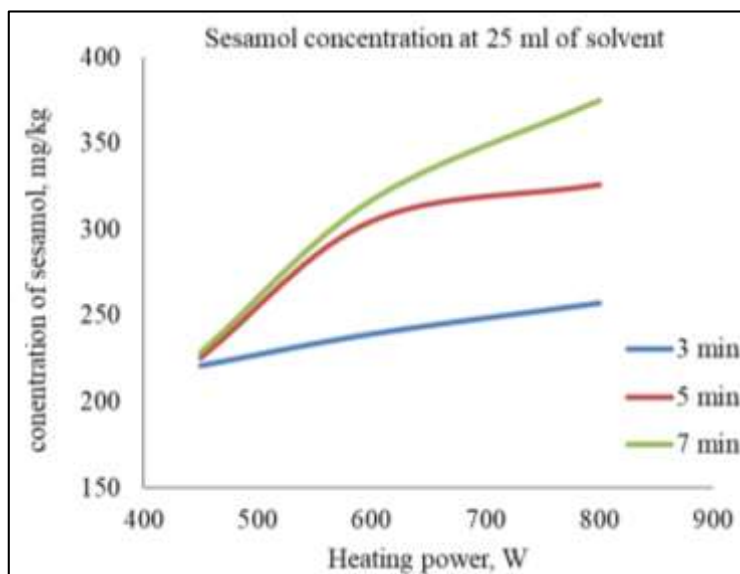


Fig 4(c): Effect of heating time and heating power on concentration of sesamol at constant solvent volume. Sesamol concentration increased as heating power and heating time were increased simultaneously.

Fig 4: Effect of different parameters on extraction yield

Conclusion

Very few research works are available on microwave extraction from 'oil' sample because of microwave heating principle itself. Polar solvent only can be used for extraction purpose but oil sample should be miscible in the solvent for homogeneous microwave heating hence, ethyl acetate worked best under given circumstances. Heating power, process time and amount of solvent volume all together affect the concentration of extracted compound. It was concluded that the concentration of extracted sesamol was reduced for combination of low solvent volume, higher power and longer processing time because at high power, solvent immediately got vaporized and it doesn't provide sufficient time to extract compound for leaching out from sample into solvent and thus at high power and long time, the solvent quantity should be increased. Therefore, with low solvent volume sample should be heated at low microwave power for longer time. Also, if higher power level is used then process time should be less to save the energy because, at higher power and sufficient solvent volume, extraction process completes very fast under microwave heating. Optimum conditions for extraction should be followed for efficient extraction process. The optimum condition for present study was observed at microwave heating of sample at 800 W of microwave heating for 7 min of processing time with 25 ml of ethyl acetate solvent, the extracted amount of sesamol from 2 g of sample at this condition was found as 374.81 mg/kg which was higher as compared to other methods such as anion exchange solid phase extraction, spectrophotometric method, colorimetric method and supercritical carbon dioxide extraction method. Maximum recovery of sesamol using anion exchange solid phase extraction from sesame oil was 307.8 mg/kg (Sun & Xiao, 2014) [17]. The same was obtained as 200 mg/kg using spectrophotometric method and colorimetric method (Suarez *et al.* 1952) [15]. Sesamol was not detected in sesame using supercritical carbon dioxide extraction methods (Date *et al.* 2011) [2].

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