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RESEARCH ARTICLE

# Focused microwave assisted extraction setup and extraction of sesamol from sesame seed

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## ABSTRACT

Microwave-assisted extraction technique opens a new room for speeding up extraction of bioactive components from food matrix while maximizing the yield recovery. In this study, a lab scale microwave assisted extraction system was fabricated which utilized focused microwave radiation. Its major components were domestic microwave oven, extraction assembly, cooling assembly and peristaltic pump. The system was used to extract sesamol from roasted sesame seed. Extraction was performed at different levels of microwave power (450, 600 and 800 W) and irradiation time (2, 4 and 6 min) with 20 ml concentrated ethyl acetate solvent. Analytical quantification was done using liquid chromatography. Extraction yield was lower at 450 W for 2 min of irradiation exposure to the sample. Maximum yield was obtained at 800 W power for 6 min of microwave heating. MAE yield was compared with soxhlet extraction method which indicated two times more recovery in case of MAE method.

**Keywords:** Focused microwave assisted extraction, soxhlet extraction, sesamol, sesame seed

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## INTRODUCTION

Microwave assisted extraction (MAE) method follows the similar extraction steps as conventional method except heating source. MAE process uses microwave heating instead of conventional heating. Microwaves are electromagnetic waves which fall in between infrared and radio waves at frequency range within 300MHz to 300GHz (Chan et al., 2011). Microwave heating has been studied as an innovative technique of heating dielectric materials. Microwaves dissipate heat within the material by the electromagnetic phenomenon of energy transfer (Kala et al., 2016). This extraction process is mainly famous in the field of medical samples, phytochemicals and environmental samples (Li et al., 2012). MAE apparatus mainly consists of a microwave oven, an extraction flask and a cooling assembly (Mandal et al., 2007). Sample is placed with polar solvent in the extraction flask which is heated and vapor gets cool and falls into extraction flask only, thus the mass transfer takes

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place from sample matrix to solvent matrix (Veggi et al., 2012). After calculated time of heating, the extract is further analyzed. When the extraction is done at atmospheric pressure then it is called as Open Vessel/focused MAE. Microwave assisted extraction process is grabbing attention towards itself because of its many advantages. Some of these are (Mandal et al., 2007; Zhang et al., 2011; Chan et al., 2012):

- It consumes less time for extraction of compound than the other methods.
- It requires less power consumption by efficient, homogenous and inbuilt heating.
- It also preserves the solvent quantity.
- MAE process preserves the thermal sensitive compounds to get denatured because it works at low temperature as compare to conventional method.
- Due to new modification in MAE like pressurized MAE, vacuum MAE, solvent free MAE etc., the extraction efficiency has been increased.

Sesame seed is the one of the oldest oil seed well known to mankind. It is originated from India. Today India is a major exporter of sesame along with China and Mexico. India produces a wide variety of Sesame seeds varying in color from white to red to black, with oil content varying from 40 to 50 % (Hemalatha, 2004). The white and black Indian Sesame seeds are mainly used for direct consumption by its addition in several foods and for exports. The brown Indian Sesame seeds are mainly used for oil extraction. These have oil content of around 45-50 %. The white Indian Sesame seeds have the desirable nutty taste and are used for making sweets, baked foods and confectionaries. The black Indian Sesame seeds are used for seasoning and for fries. Hulled seeds are also used for snack preparation. Sesamol is one of compound found in sesame seed which increases the self-life of seed (Hwang, 2005). It also acts as anti-carcinogenic compound which helps to prepare anti-cancer medicines.

Therefore, this study focuses on extraction of sesamol from roasted sesame seed. Prior to extraction process, lab scale MAE system was fabricated. Further, MAE extraction yield was compared with extract using soxhlet extraction method.

## MATERIALS AND METHODS

### Set up of lab scale focused MAE system

#### *Selection of microwave heating system*

The main component of MAE apparatus was microwave oven on which the complete process was dependent. Therefore, selection of Microwave oven was very crucial step. Selection was based on maximum output power of microwave oven, mode of microwave heating, type and size of microwave oven and maximum temperature within microwave oven (Ericsson and Colmsjö, 2000). In general, microwave oven used at domestic level has maximum power as 800 W. So we fixed the maximum output power as 800 W. Size of cavity or capacity of microwave oven was selected according to extraction assembly put inside the cavity. Thus the length of microwave oven cavity was estimated as minimum 200 mm. which was found in 21 L capacity microwave oven. Therefore, we selected Samsung CE74JD convective type microwave oven for our research purpose that had 800 W maximum output power at microwave mode and 1100 W at convection mode with 6 power levels and 200˚C as maximum temperature.

***Selection of extraction assembly***

Extraction assembly was chosen on the basis of its size, bottom type and number of openings. In general, MAE process consumes maximum 50 ml of solvent. So 250 ml of round bottom flask was chosen. Side opening of flask gave an additional benefit of continuous supply of solvent. So 250 ml, side opening, round bottom flask made up of borosil glass was chosen as extraction flask.

***Selection of cooling assembly***

Cooling assembly was attached to the extraction flask to condensate the vapor formed by microwave heating. For that a condenser was selected on the basis of type of condenser and length of condenser. Graham glass condenser was chosen because it has more cooling surface area which gives more efficient cooling than other type of condenser. Length of condenser was chosen by heat and mass transfer calculation (Pare, 2010). This calculation was performed for ethanol which is a frequently used solvent in MAE process.

Heat realized by vapor formed during microwave heating = Heat gain by cooling water flowing in condenser

Heat required for cool down the temperature of vapor from $T_1$ to $T_2$ = Heat required to raise the cooling water temperature from $T_1'$ to $T_2'$

$$mc_p \Delta T + m\lambda = UA\Delta T_{LMTD}$$

where, $T_1$ = temperature of solvent vapor at entry point of condenser (80°C)

$T_2$ = temperature of condensed solvent (50°C)

$T_1'$ = temperature of water at inlet of condenser (20°C)

$T_2'$ = temperature of water at outlet of condenser (45°C)

(Temperatures are selected on the experiments basis)

m = mass of solvent vapor (assumed as maximum solvent flow rate as 50 ml/min)

$c_p$ = specific heat of solvent (here solvent was ethanol so $c_p$ = 2.430 J/g /°C)

$\Delta T$ = decrease in temperature of solvent raised by cooling water

$\lambda$= latent heat of condensation ($846\times10^3$ J/kg for ethanol)

U = over all heat transfer coefficient (1135.72 Watt/$m^2$K for graham condenser)

A = surface area of condenser coil

$\Delta T_{LMTD}$ = log mean temperature difference

$=(\Delta T - \Delta T')/ \log (\Delta T/ \Delta T')$

$= (35-30)/\log (35/30)$

$= 32.44^{o}C$

$m = 50\times10^{-3}\times10^{-3}\times789$ kg/min $= 39.450\times10^{-3}$ kg/min

$mc_p \Delta T + m\lambda = UA \Delta T_{LMTD}$

$39.450\times10^{-3}(2430\times28.5+846000)/60 = 1135.72\times A\times32.44$

$A = 0.0164$ m$^2$

Where, $A = \pi DL$, D is coil dia (8 mm) and L is coil No of turns in coil (n) = 21 (assumed)

Thus, the length of condenser = n × distance b/w two turns

$L = 21\times14$

$= 294$ mm

Where, L is the length of condenser

Thus, 300 mm length graham condenser was used for MAE apparatus (Fig 2). Water chiller was attached to the condenser for efficient cooling of the vapor formed during microwave heating.

***Selection of pump***

This is an additional feature attached to the MAE system used for continuous supply of solvent. So, peristaltic pump was attached to MAE apparatus. Peristaltic pump has 100 cc/h maximum flow rate. This pump was attached to MAE apparatus by silicon tube having inner diameter as 1cm and thickness as 0.5 cm.

**Raw materials**

Extraction was performed from roasted and ground sesame seed. Black sesame seed was procured from local market of Kharagpur, West Bengal. Sample was prepared as shown in fig 1. Ethyl acetate was used as extraction solvent. All the chemicals were procured from lab solution, Kolkata, West Bengal. 5 g of 98% pure sesamol was purchased from Sigma, India.

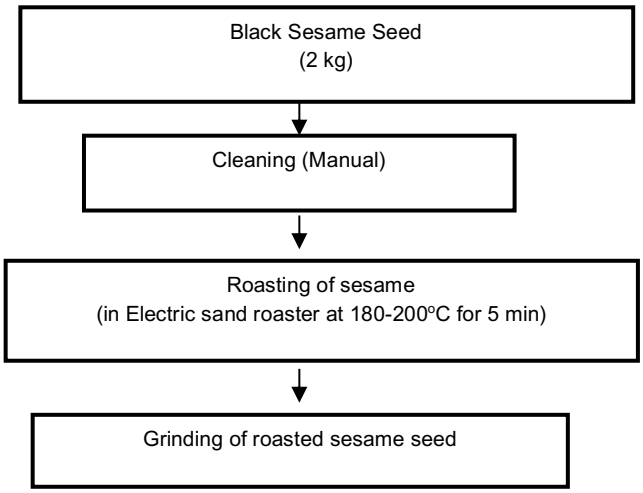


**Fig. 1 Sample preparation procedure**

**Microwave assisted extraction process**

MAE process of sesame seed was performed at different microwave oven output power and heating time. MAE of sample was executed as per earlier described method by Maran et al. (2013). MAE experiment design was done as per factorial design method. For factorial, 3 variables and the range of these three variables are as microwave power 450-800 W and process time 2-6 min was taken according to trials. Concentration of ethyl acetate was 99.5%. In case of sesame seed, a little modification was done for improving homogenous microwave heating during irradiation because some of the solid particles stuck to surface of extraction flask and did not get homogenous heating. So, after each 2 min heating 1 min was allotted for stirring. So the actual time taken for 2 min heating was 3 min (1+2), 4 min heating was 6 min (1+2+1+2) and for 6 min heating it was 9 min (1+2+1+2+1+2). Design was done according to $N= L^V$ (N is total numbers of experiments, L is number of levels (3) and V is number of variable factors (2))

**Table 1. $+a_m$, 0 and $-a_m$ values for sesame seed sample**

| $+a_m$ | 0 | $-a_m$ |
|---|---|---|
| **Calculated values** | | |
| 800 W | 625W | 450 W |
| 6min | 4 min | 2 min |
| **Actual values taken for experiment** | | |
| 800 W | 600 W | 450 W |
| 6 min | 4 min | 2 min |

**Soxhlet extraction process**

Soxhlet extraction method has performed according to Suja et al. (2004). 5 g of defatted sesame seed put into the thimble with 250 ml of methanol in flask at 80°C for 16 hrs. Extract was filtrated with watt man filter paper 4 and kept in refrigerating condition till the further use.

### High performance liquid chromatography process

For analytical measurement of sesamol HPLC protocol given by Suja et al. (2004) was used. HPLC was performed at central research facility, IIT Kharagpur. Agilent 1100 series with a UV detector and C-18 column thermostated at 35°C was used for sesamol analysis. Filtered samples were injected with mobile phase of 70% of methanol at the flow rate of 1 ml/min. Sesamol was detected by the absorbance at 290nm. Calibration curve was obtained by using five different concentration of standard solution of sesamol (10, 50, 100, 200 and 500 mg/L) prepared by serial dilution of stock solution 5 g/L. Sesamol was quantified according to the calibration curve (Suja et al., 2004).

## RESULTS AND DISCUSSION

### Set up of lab scale focused MAE system

MAE system was designed as mentioned earlier and sent for the fabrication to the Lab Solution, Kolkata. Setup consisted CE74JD Samsung microwave oven, 250ml round bottom, side opening extraction flask, and 300 mm graham condenser with the fittings for pump. Fabricated system has been shown in fig 2 and fig 3. The output power level could be varied from 80 to 800. After required microwave exposure, the mixture was filtered and sesamol was quantified using HPLC method.

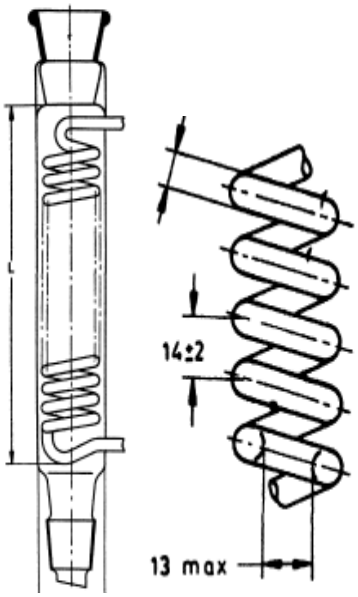


**Fig. 2: Cross sectional view of condenser**

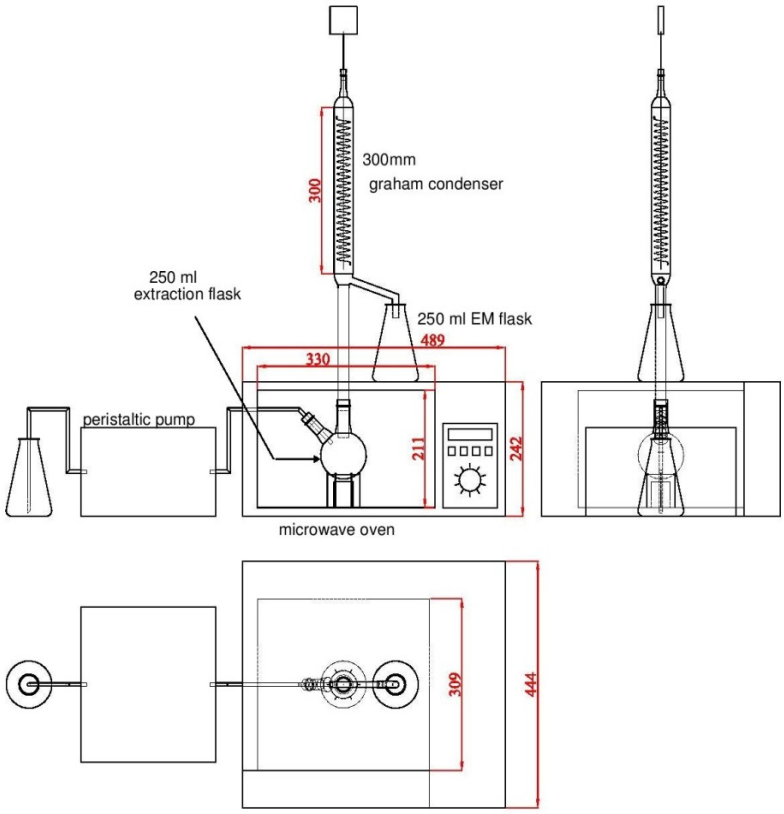


**Fig. 3 Two-D design of focused MAE apparatus (all dimensions in mm)**

**Quantification of sesamol microwave assisted extraction process**

MAE process was done at different combinations of independent variables which included process time (2-6 min) and irradiation power (450-800 W). According to the factorial design method, 9 experiments were conducted. Due to microwave heating of sample-solvent mixture, sample matrix got broken down and compounds from sample were transferred to the solvent matrix by leaching and got dissolved in solvent. This solvent mixture was collected and after filtration it was kept at refrigeration condition till further use.

Quantity of sesamol in sesame seed was found as 298.6 mg/kg of sample at 800 W for 6min of irradiation heating (Table 2). According to studies, the quantity of sesamol in sesame seed is higher than the sesame oil because during oil extraction process, some amount of sesamol gets lost (Hwang, 2005), Here, we saw that sesamol amount in sesame seed is less as compare to sesame oil (data not shown). It is because of incomplete breakdown of sample matrix. By the Soxhlet extraction, maximum yield recovery of sesamol was 128.3467 mg/kg. It indicates approximately two times more recovery by MAE process than the soxhlet extraction process.

**Table 2: Concentration of sesamol in sesame seed sample (mg/kg)**

| Run | Different MAE operating conditions | | Retention time, min | Concentration of sesamol in sample, mg/kg |
|---|---|---|---|---|
| | Power, W | Time, min | | |
| $S_1$' | 450 | 2 | 2.029 | 70.2±1.5 |
| $S_2$' | 450 | 4 | 2.015 | 148.6±1.3 |
| $S_3$' | 450 | 6 | 2.100 | 259.5±0.5 |
| $S_4$' | 600 | 2 | 2.031 | 133.5±0.7 |
| $S_5$' | 600 | 4 | 2.070 | 144.7±0.9 |
| $S_6$' | 600 | 6 | 2.006 | 279.84±0.3 |
| $S_7$' | 800 | 2 | 1.998 | 134.8±1.0 |
| $S_8$' | 800 | 4 | 2.000 | 157.8±1.7 |
| $S_9$' | 800 | 6 | 2.034 | 298.6±0.7 |

## CONCLUSION

Maximum recovery of sesamol was obtained at 800 W irradiation power for 6 min of extraction time with 20 ml of ethyl acetate solvent. Maximum recovery was 298.6 mg/kg. By comparing the MAE yield with soxhlet extraction yield, we found two times more yield recovery by MAE process than the soxhlet extraction process. Microwave assisted extraction was currently regarded as a robust alternative to traditional extraction techniques, especially in the case of the sample preparation for analytical purpose. It saves process time, power consumption, solvent volume and it is economically efficient.

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