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MICROWAVE ASSISTED PROCESS (MAP) FOR EXTRACTION OF FRAGRANCES FROM SELECTED MALAYSIAN FLOWER

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ABSTRACT

The Microwave Assisted Process (MAP) is a high-speed method used to selectively extract target compounds from various raw materials. It was originally developed for the extraction of flavors and fragrances from plant materials, which is generally carried out by techniques that require a lot of energy or a long time or a combination of both. In this study, the important process parameters; the time, temperature and microwave power of the microwave extraction system are controlled in order to obtain the highest yield of extracted essential oil in less time. The Microwave Extraction and Conventional Method Extraction are compared for the extraction of essential oil from Jasmine flowers (*Jasminum officinale*). The Microwave Extraction Method provides more valuable essential oils, reduces the extraction time and allows a substantial saving of energy. After 60 minutes of Microwave Extraction, it is possible to collect sufficient essential oil for analytical determinations, whereas Conventional Method Extraction requires 8 hours. Gas chromatography-mass spectrometry (GC-MS) is used for detection and identification of the extracted compounds. The composition of essential oils isolated by Microwave Extraction and Conventional Method Extraction show a major difference in terms of their aromatic profiles.

INTRODUCTION

Extraction or separation represents a change in the relative concentrations of two or more sample components within a defined region, as a result of the transfer of chemical species (components) from one region to another. There are many extraction methods such as the Soxhlet, Separatory Funnel, Sonication, Supercritical Fluid (SFE), Accelerated Solvent Extraction (ASE) and Microwave Extraction. These conventional methods require large volume of solvent, long runtime, high energy consumption, high temperature / pressure conditions which leads to production of artefacts, high operational costs which is suitable only for a limited group of high value-added target materials and not representative of large-scale treatment process or does not have industrial-scale equivalent.

A few years ago, the Microwave Assisted Process (MAP) was introduced for the extraction of essential oils and other high-value substances from a broad range of

matrices. Essential oil is the fragrant principle of a plant. They are chemical components that give a plant its characteristic fragrance. Essential oils are currently being extracted from natural products either by hydrodistillation or solvent extraction. Losses of some volatile compounds, low extraction efficiency, degradation of unsaturated compounds through thermal or hydrolytic effects and toxic solvent residue in the extract may be encountered using these extraction methods. These shortcomings have led to the consideration of the use of microwave-assisted process in essential oil extraction processes. This process allows for direct extraction of fresh material without the need to dry them prior to the extraction, as commonly being the prerequisite in most other methods. This MAP overcomes some of the major limitations of current extraction remediation technologies, such as the requirement for long extraction times and large volumes of solvent, while increasing the extraction efficiency.

Following the results of the various experiments carried out it becomes obvious that extraction using microwave technology is a good alternative to conventional extraction techniques. The extraction by microwave as a new extraction technique has its own specific parameters that need to be characterized for every plant that contains essential oil. Conditions are important criteria and the microwave generator have to be designed in such a way that the most important process parameters can be adjusted and controlled throughout the process [1]. In this study for the extraction of essential oil from Malaysian flower, the Milestone Ethos SEL Microwave Labstation, which is a computerized microwave extraction system, is used for the instrumentation work. Lab terminal 800 Controller was supplied with the microwave system for overall operation control [4]. It comes with a control software which allows the user to change the microwave parameters; the time, temperature and microwave power during the run in order to obtain the maximum yield of extracted essential oil in less time, which allows for real-time optimization during method development.

MATERIALS AND METHOD

The vast majority of true essential oils are produced by distillation. There are different processes used, however. In all of them, water is heated to produce steam, which carries the most volatile chemical of the aromatic material with it. The steam is then chilled in a condenser and the resulting distillate is collected. The essential oil will normally float on top of the distilled water component and maybe separated off. In this study for the extraction of essential oil from Jasmine, three methods of extracting essential oil; dry distillation, hydrodistillation and steam distillation method are used in the microwave extraction and conventional method extraction.

Microwave Extraction
Microwave extractions are performed in the Milestone Ethos SEL Microwave Labstation, operating at 2455 MHz with a maximum delivered power of 1000W, variable in 10W increments. The dimensions of the PTFE-coated cavity are 35 x 35 x 35 cm. The time, temperature and microwave power control are adjusted and controlled throughout the process using the Lab terminal 800 Controller. Temperature is monitored

with the aid of a fiber-optic temperature sensor. In a typical Microwave Extraction (SFME) procedure which combines microwave heating and dry distillation, 200g of Jasmine flowers are put in a 2 liter round bottom flask and placed in the microwave reactor without any added solvent or water. As for the hydrodistillation method, the flowers are fully submerged in water but for steam distillation method, a prototype model, which uses an outside source of steam, which pipes the steam into the distillation unit, will be designed. These experiments are performed at 450W, 500W and 550W for 60, 70, 80 and 90 minutes. The first essential oil droplet in each extraction is recorded. The essential oil is collected, dried with anhydrous sodium sulphate and stored at 0^{o}C until used [3].

Conventional Method Extraction
For Conventional Method Extraction, the jasmine flowers are submitted to hydrodistillation and steam distillation with a Clevenger-type apparatus according to European Pharmacopoeia consisting of a 2 liter round bottom flask set in a heating mantle and extracted at 450W for 8 hours until no more essential oil is obtained. The first essential oil droplet is also recorded. The essential oil is then collected, dried with anhydrous sodium sulphate and stored at 0^{o}C until used [3].

Gas Chromatography-Mass Spectrometry Identification
The essential oils are analysed by GC-MS (Hewlett-Packard computerized system comprising a 5890 gas chromatograph coupled to 5971A mass spectrometer) using a fused-silica capillary column with an apolar stationary phase SBP5TM (60m x 0.32mm i.d., 1 μm film thickness). GC-MS are obtained using the following conditions: carrier gas, He; flow rate, 1 ml/min; split, 1:20; injection volume, 0.1 μm; injection temperature, 250 ^{o}C; oven temperature programme, 60-200 ^{o}C at 4 ^{o}C/min, then held at 200 ^{o}C for 30 min; the ionization mode used is electronic impact at 70 eV. Identification of the components is achieved from their retention indices on both columns, determined with reference to a homologous series of alkanes, and by a comparison of their mass spectral fragmentation patterns with those stored in the data bank (Wiley/NBS library) and the literature.

The amount and the composition of the essential oil obtained by both Microwave Extraction and Conventional Method Extraction are compared for further analysis. The physical and chemical constants of the essential oil such as the colour, aroma, taste, specific gravity, refractive index and optical rotation can be obtained where these characteristics are important in the determination of the quality of an essential oil. A prototype model which combines microwave heating and steam distillation will be developed for large-scale treatment.

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RESULTS AND DISCUSSION

The extraction time, yield, and the details of the microwave extraction are shown in Table 1.

Table 1: Details of Wet Jasmine Extraction

MICROWAVE EXTRACTION					CONVENTIONAL EXTRACTION				
Power (Watt)	**Temp (^{o}C)**	**Time Of Ext (Min)**	**Time Of First drop (Min)**	**Yield (g)**	**Power (Watt)**	**Temp (^{o}C)**	**Time Of Ext (Min)**	**Time Of First drop (Min)**	**Yield (g)**
450	100	60	00:9:10	0.1	450	100	8hours	00:15:30	0.1
	100	70	00:8:45	0.1					
	100	80	00:8:25	0.1					
	100	90	00:8:46	0.1					
500	100	60	00:7:14	0.1					
	100	70	00:7:29	0.1					
	100	80	00:7:20	0.1					
	100	90	00:7:26	0.1					
550	100	60	00:6:28	0.1					
	100	70	00:6:46	0.1					
	100	80	00:6:12	0.1					
	100	90	00:6:24	0.1					

Table 2: Details of Dry Jasmine Extraction

MICROWAVE EXTRACTION				
Power (Watt)	**Temperature (^{o}C)**	**Time Of Extraction (Min)**	**Time Of First Drop (Min)**	**Amount Of Yield (g)**
450	100	60	3:38	0.1
	100	70	3:53	0.1
	100	80	4:10	0.1
	100	90	4:02	0.1
500	100	60	3:32	0.1
	100	70	3:38	0.1
	100	80	3:12	0.1
	100	90	3:20	0.1
550	100	60	3:18	0.1
	100	70	3:21	0.1
	100	80	3:00	0.1
	100	90	2:54	0.1

Table 3: Details of Steam Jasmine Extraction

CONVENTIONAL EXTRACTION				
Power (Watt)	**Temp (^{o}C)**	**Time Of Ext (Min)**	**Time Of First Drop (Min)**	**Yield (g)**
450	100	8 hours	01:42:00	0.1

Below are the heating profiles for Wet Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 450W, 100°C)

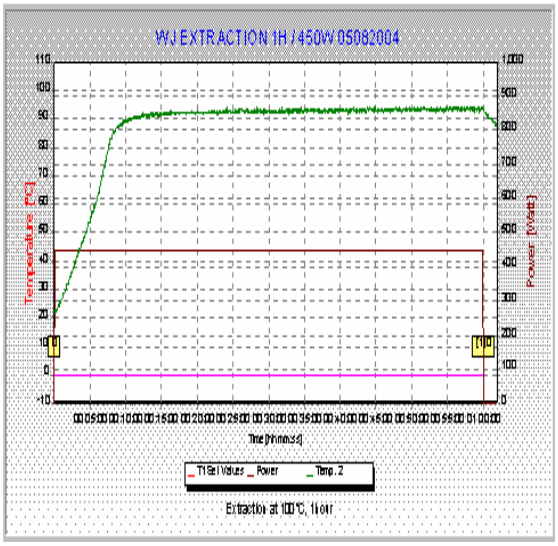

Figure 1: Parameter profile for extraction at 450W, 60 minutes, 100°C

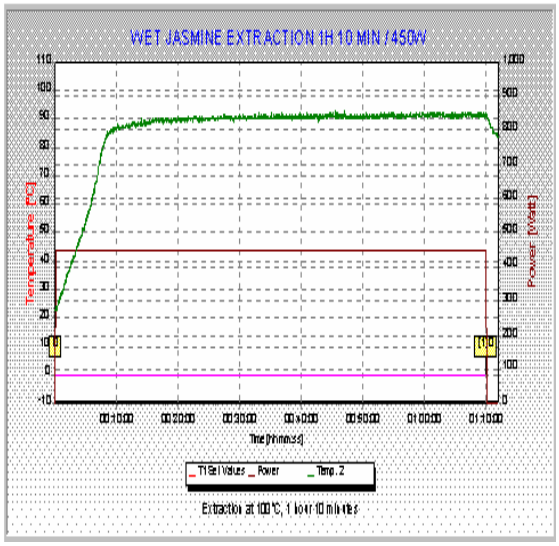

Figure 2: Parameter profile for extraction at 450W, 70 minutes, 100°C

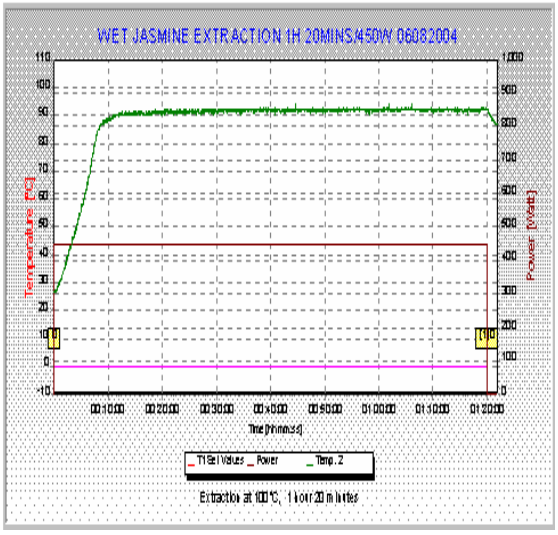

Figure 3: Parameter profile for extraction at 450W, 80 minutes, 100°C

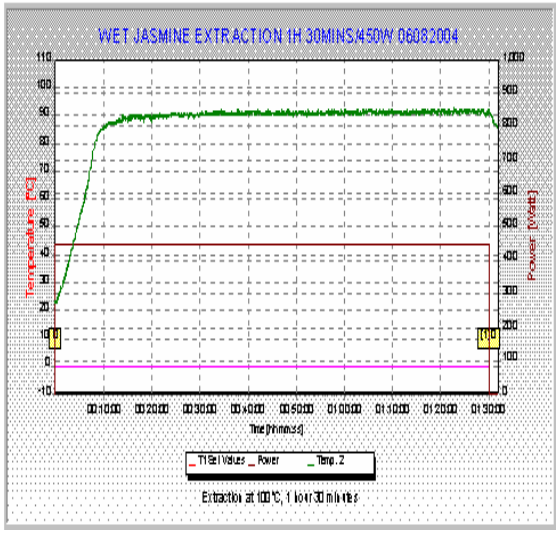

Figure 4: Parameter profile for extraction at 450W, 90 minutes, 100°C

Below are the heating profiles for Wet Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 500W, 100°C)

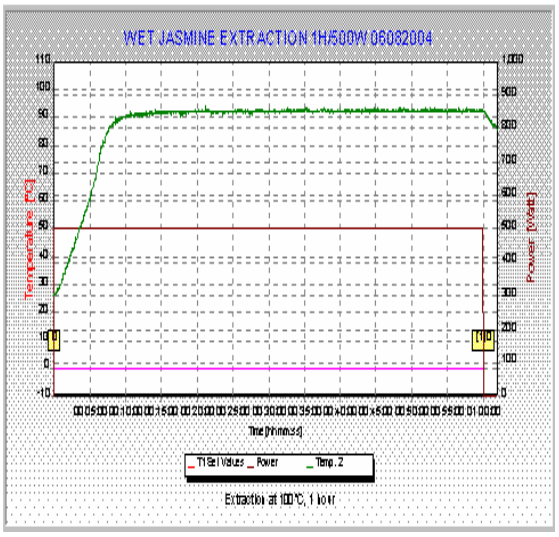

Figure 5: Parameter profile for extraction at 500W, 60 minutes, 100°C

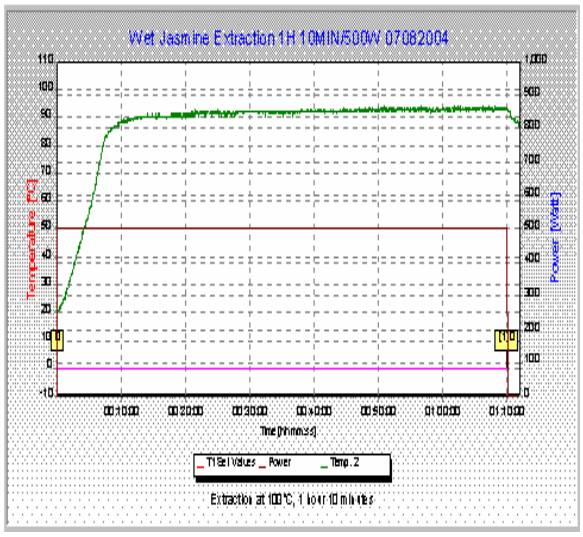

Figure 6: Parameter profile for extraction at 500W, 70 minutes, 100°C

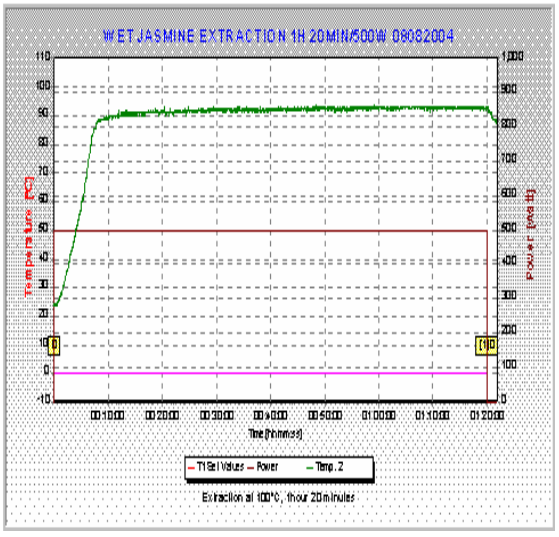

Figure 7: Parameter profile for extraction at 500W, 80 minutes, 100°C

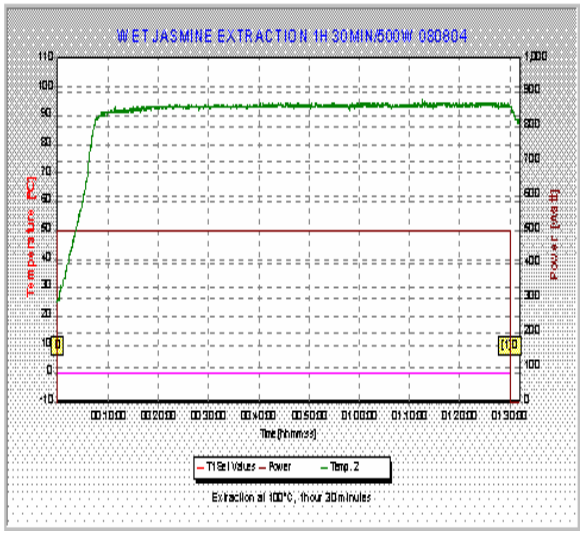

Figure 8: Parameter profile for extraction at 500W, 90 minutes, 100°C

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Below are the heating profiles for Wet Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 550W, 100°C)

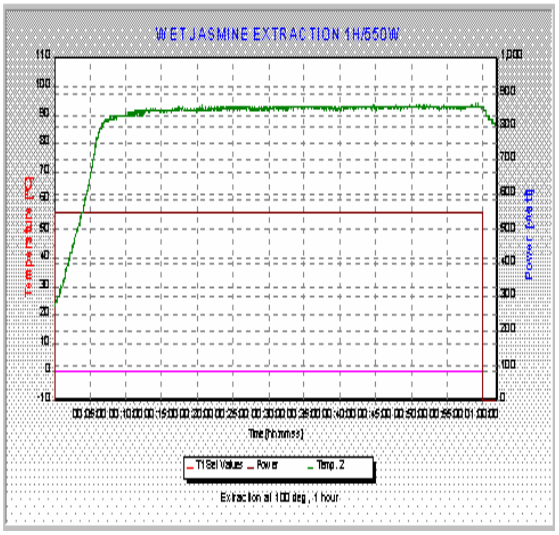

Figure 9: Parameter profile for extraction at 550W, 60 minutes, 100°C

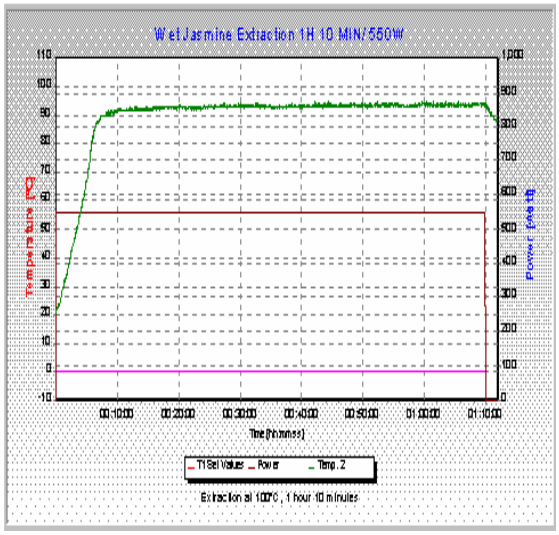

Figure 10: Parameter profile for extraction at 550W, 70 minutes, 100°C

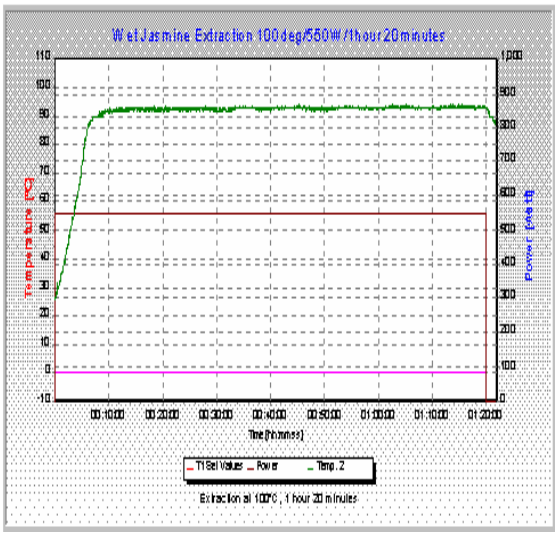

Figure 11: Parameter profile for extraction at 550W, 80 minutes, 100°C

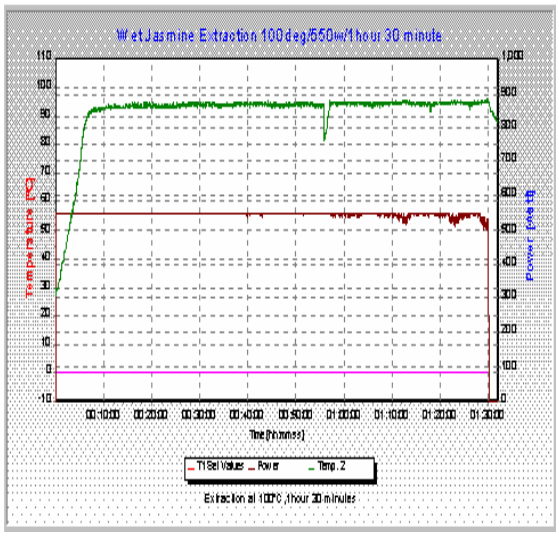

Figure 12: Parameter profile for extraction at 550W, 90 minutes, 100°C

Below are the heating profiles for Dry Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 450W, 100°C)

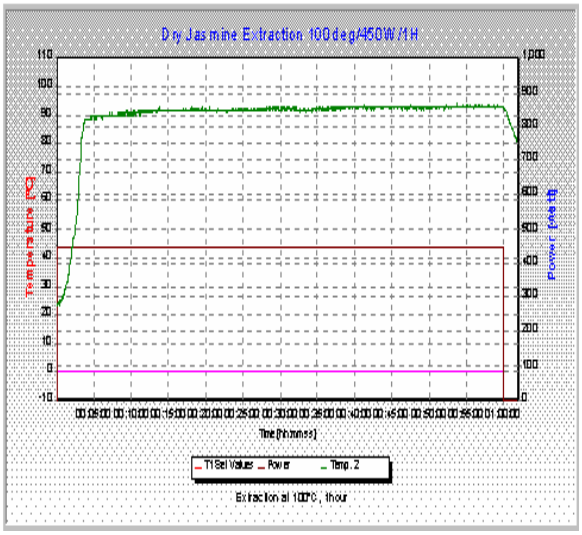

Figure 13: Parameter profile for extraction at 450W, 60 minutes, 100°C

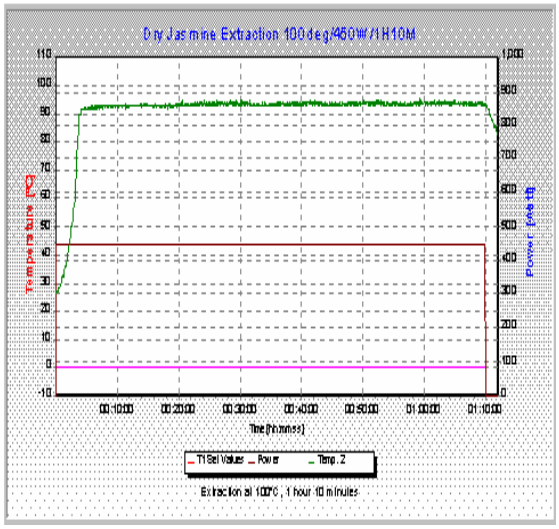

Figure 14: Parameter profile for extraction at 450W, 70 minutes, 100°C

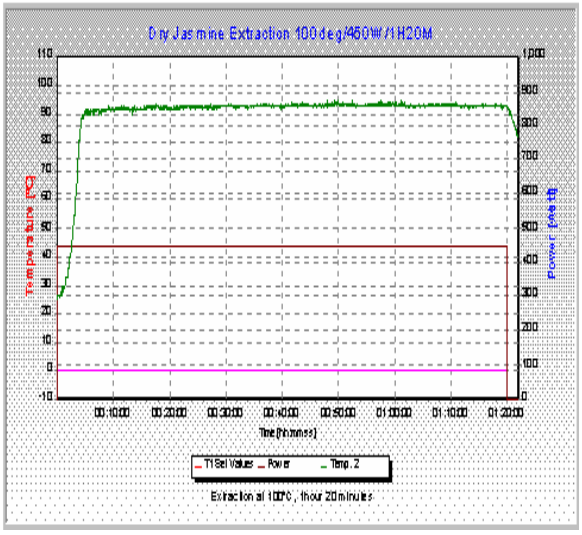

Figure 15: Parameter profile for extraction at 450W, 80 minutes, 100°C

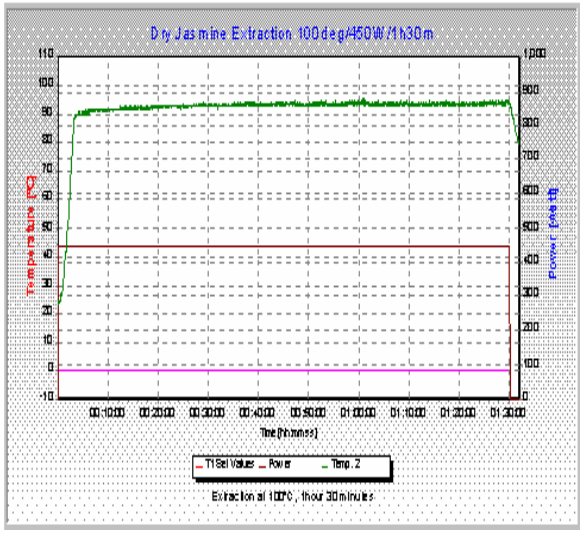

Figure 16: Parameter profile for extraction at 450W, 90 minutes, 100°C

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Below are the heating profiles for Dry Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 500W, 100°C)

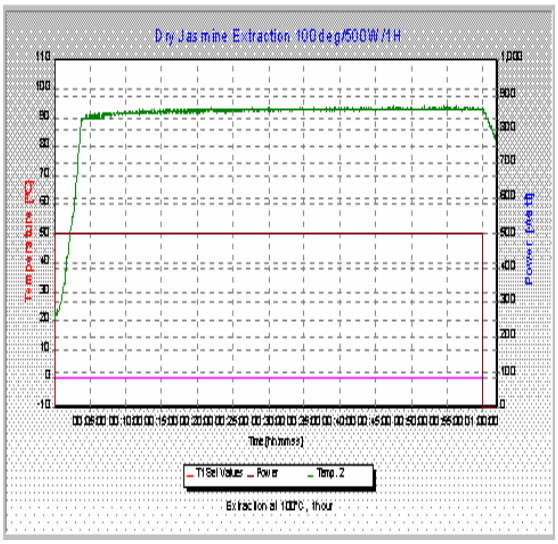

Figure 17: Parameter profile for extraction at 500W, 60 minutes, 100°C

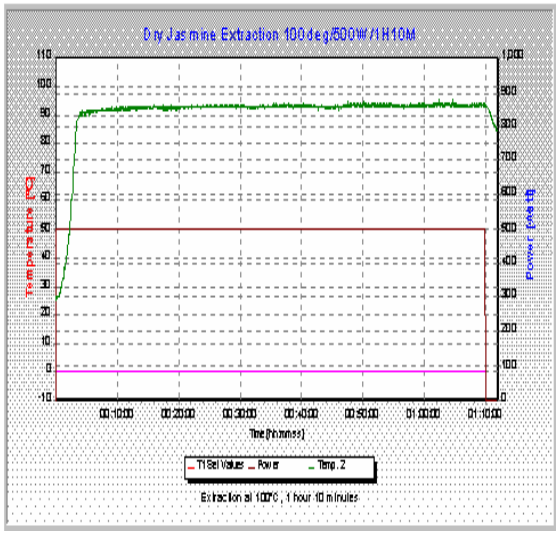

Figure 18: Parameter profile for extraction at 500W, 70 minutes, 100°C

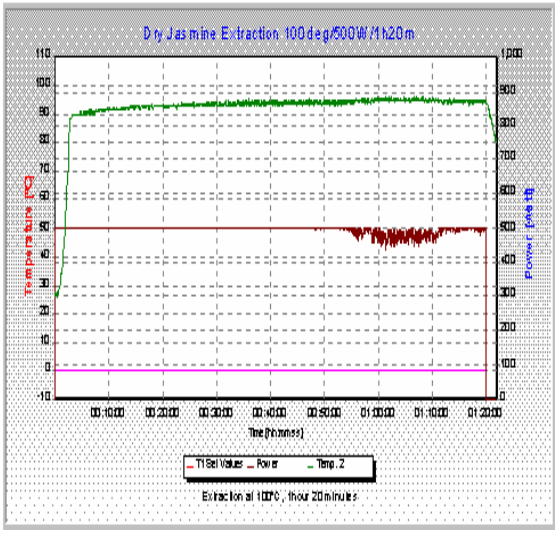

Figure 19: Parameter profile for extraction at 500W, 80 minutes, 100°C

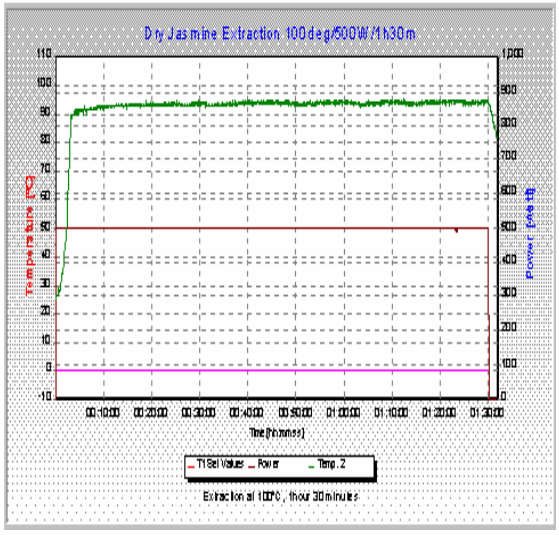

Figure 20: Parameter profile for extraction at 500W, 90 minutes, 100°C

Below are the heating profiles for Dry Jasmine Extraction using Microwave Ethos SEL Labstation (60-90 minutes, 550W, 100°C)

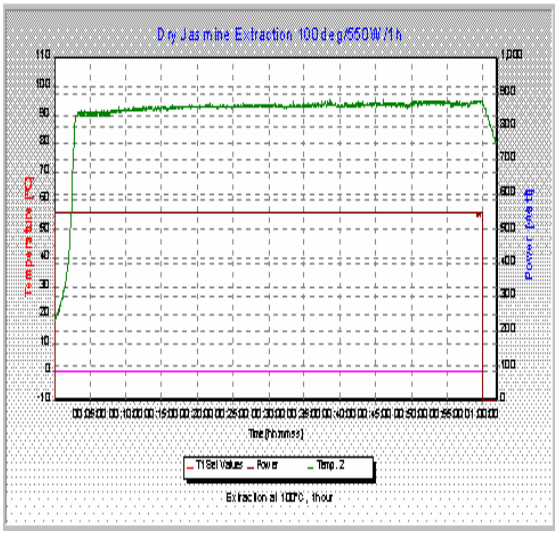

Figure 21: Parameter profile for extraction at 550W, 60 minutes, 100°C

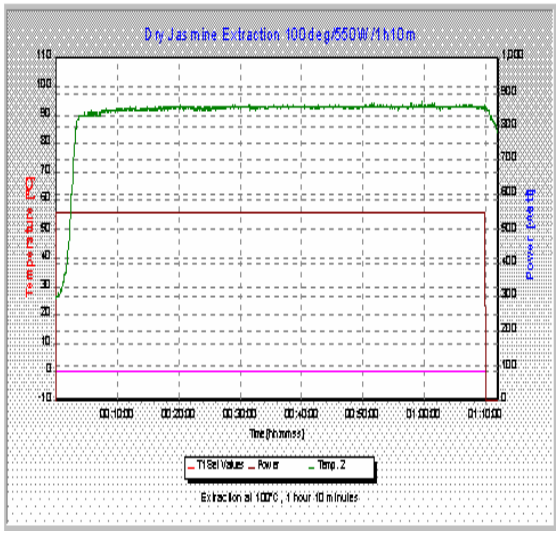

Figure 22: Parameter profile for extraction at 550W, 70 minutes, 100°C

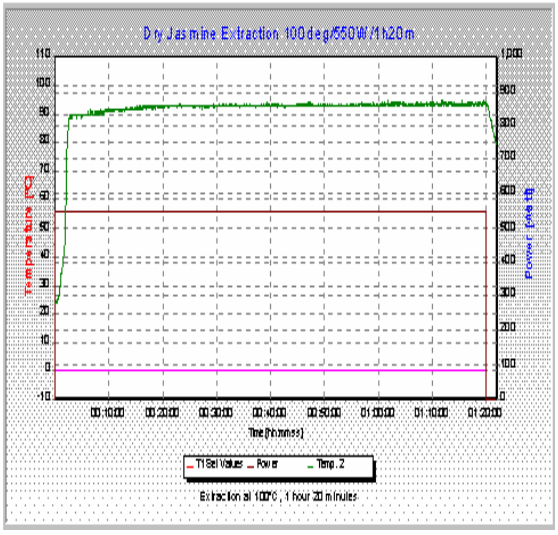

Figure 23: Parameter profile for extraction at 550W, 80 minutes, 100°C

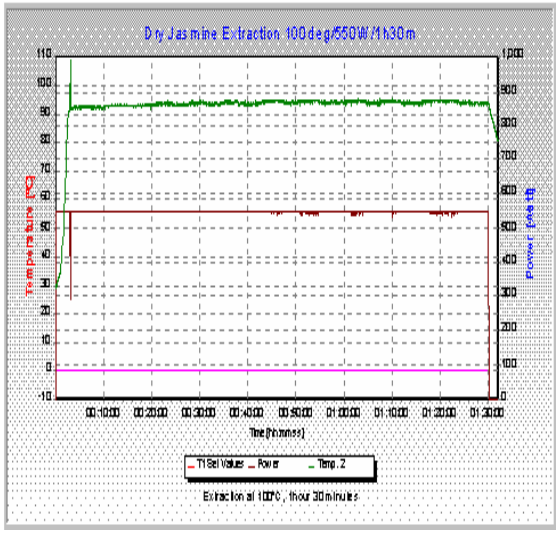

Figure 24: Parameter profile for extraction at 550W, 90 minutes, 100°C

One of the advantages of the Microwave Extraction is rapidity. For Conventional Method or Microwave Extraction, the extraction temperature is equal to water boiling temperature at atmospheric pressure (100°C). To reach the extraction temperature

(100^{o}C) and thus obtain the distillation of the first essential oil droplet, it is necessary to heat only 3-4 minutes for Dry Method and 6-9 minutes for Wet Method using Microwave Ethos SEL against 15 minutes for Wet Method and 90 minutes for Steam Method using Heating Mantle. After 60 minutes of Microwave Extraction, it is possible to collect sufficient essential oil for analytical determinations, whereas Conventional Method Extraction requires 8 hours. The Microwave Extraction method also allows a substantial saving of energy. 60-90 minutes of Microwave Extraction at 450W, 500W and 550W for both Wet and Dry Method give the same amount of yield that is 0.1g as the 8 hours of Conventional Extraction for Wet and Steam Method.

However, the composition of essential oils isolated by Microwave Extraction and Conventional Method Extraction show a major difference in terms of their aromatic profiles. There are over 100 constituents in the oil including benzyl acetate, linalool, phenylacetic acid, benzyl alcohol, farnesol, methyl anthranilate, cis-jasmone, methyl jasmonate, among others [2]. However, there are slightly fewer compounds in the essential oil extracted by Microwave Extraction compared with that obtained by Conventional Method Extraction. Indeed, some minor compounds are present only in essential oils isolated by Conventional Method Extraction but substantial higher amounts of highly odoriferous compounds, which are more valuable, are present in the Microwave Extraction extract. The loss of some compounds in the Microwave Extraction is not an artefact of the extraction. The reduction of extraction time and the amount of water used in Microwave Extraction method necessarily reduce the deterioration of principal compounds by thermal and hydrolytic reactions (hydrolysis, trans-esterification or oxidation) and the generation of degradation products. Water is a polar solvent that accelerates many reactions, especially reactions via carbocation as intermediates [3].

CONCLUSION

The Microwave Extraction method provides more valuable essential oils, reduces the extraction time, and allows a substantial saving of energy. After 60 minutes of Microwave Extraction, it is possible to collect sufficient essential oil for analytical determinations, whereas Conventional Method Extraction requires 8 hours. Substantial higher amounts of highly odoriferous compounds are present in the Microwave Extraction extract. Being highly aromatic, the Jasmine essential oils are prized in the perfume industry and the scent also plays an important role in aromatherapy. They are used for many different reasons and in a number of different ways. The uses and aromatherapy applications of Jasmine essential oils are as varied and adaptable as the widely planted trees themselves. Medicine, industry, perfumery and of course aromatherapy have all come to rely on the properties of Jasmine oil and are likely to do so in the future. Following the results of the various experiments carried out it becomes obvious that extraction using microwave technology is a good alternative to conventional extraction techniques especially for the extraction of essential oil for analytical determinations and for perfumery and aromatherapy purposes.

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