Analysis of Conventional and Microwave Assisted Technique for the Extraction of Concentrated oils from Citrus Peel

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Abstract— Citrus peel belongs to orange and lemon plant that contains concentrated oils. Oil from citrus peel is widely used in foods, perfumes and pharmaceutical industry worldwide. In this study, the advantages of citrus concentrated oil extracted by conventional and novel techniques were studied. Microwave extraction techniques have come out as new alternatives to conventional techniques (hydro distillation) for extraction of oils. This paper reviews the novel separation technique with the conventional techniques in terms of extraction time, yields and energy. Extraction of oils with solvent free microwave extraction (SFME) was comparatively better in terms of extraction time that is 50 minutes while for microwave assisted hydro distillation (MAHD) is 60 minutes and for hydro distillation (HD) it is 3 hours. Yields percentage was almost same for the three processes that are 1.67%. Energy savings were greater in both MAD and SFME that is 0.4 kWh while in Hydro distillation it is 1.3 kWh. Overall MAD was better in performance than other techniques.

Keywords: Citrus Peels essential oil, Hydro Distillation, Microwave Assisted Distillation, Solvent-Free Microwave Extraction.

I. INTRODUCTION

Citrus fruits including types of oranges and lemons are largely grown in different regions of the world [1]. It is one of the major crops in Pakistan. The annual production of citrus fruit in Pakistan is about 2000 k. tons [2] while global production of lemon is about 7.3 million tons and that for oranges are 2.4 million tons [3]. Citrus fruits are largely consumed for juice. Besides these conventional methods, solvent extraction is also used for extracting essential oils. In the experiment of Lopresto et. al. found that a yield of 0.95% (v/w%) was observed for Lemonene through solvent extraction in Soxhlet apparatus. Hexane was used as solvent [13]. Other solvents used for extraction of oils from citrus peels include: Methyl Alcohol, Methylene Chloride, Etyh Alcohol and Acetone etc. [14-17]. These conventional techniques provide good basis for extraction but on expense of energy and time.

Here in this research article experiments are done for the comparison and analyzing microwave assisted distillation (MAD) with the conventional techniques. The main difference is in heating mechanism. In microwave heat in the molecules are generated through waves, these waves vibrate the molecules and thus heat is produced so in MAD heat generation takes place from inside of body while in the steam and hydro distillation heat transfer occurs from out to inside of the body [18].

Extraction so it leaves a lot of waste in the form of its peels. About 50% of wet fruit waste is of citrus peels [4]. These waste products are used to feed animals or a major portion of it is directly dumped into open-air, which causes adverse effects on the environment. Citrus fruits are rich in vitamin C, carotenoids and bioactive compounds [5].

Citrus peels are rich in essential oils that contain some significant compound good for skin and health and are widely used for flavoring, in perfumery, cosmetics, medicine and pharmaceuticals [6]. Many essential compounds can efficiently be extracted from citrus peels. Citrus peels have bio-active phenols i-e flavonoids and phenolic acids. These phenols have significant properties like antioxidants, antiviral, antimicrobial, anti-inflammatory and neuroprotective, etc. [7]. Therefore, it is very important to use this waste to extract these valuable compounds from it.

The extraction of essential oils from citrus peels is done conventionally by two methods. 1. Hydro distillation and 2. Steam Distillation. In hydro distillation, dry peels are subjected to heat along with some ratio of water in a specific Clevenger type apparatus for several hours. Water and essential oils evaporate out and then condensed through the condenser in a separate flask where it is left for phasing out from water and thus collected for further analysis. Salma et. al. in their experiments found that after 4 hours extraction of essential oils from clementine the yield obtained was about 0.73% (v/w%) [8]. In one other experiment by Mohamed et. al. obtained yield of 0.24% (v/w%) from 3-hour long hydro distillation [9]. 0.42% (w/w%) yield was obtained in similar experiment performed by Uysal et. al. [10].
Steam distillation is still widely used for extraction of oils. In its steam is passed through the material from which oils are to be extracted and the rest of the process is same as of hydro distillation. Some times third solvent is used for layer separation of extracted oil from water and then heat treatment is done to vaporize the concentrated oil leaving behind. In one such experiment performed by Pauline et. al. They extracted oil by steam distillation in two-hour long process and at temperature of just above 100 oC, then sodium chloride and chloroform were added to the separating funnel and shaken back and forth several times and thus layer separation occurs. A yield of 2.475% (w/w%) was observed [11]. Kusuma et. al. obtained a yield of 0.59% (v/w%) by steam distilling citrus peels for 7 hours [12].

II. MATERIALS AND METHODS

Citrus Limonum peels were used for extraction of oil along with water. Sodium sulphate was used to remove any water present in essential oils.

For hydro distillation Clevenger type apparatus was used. 100 g of citrus peels cut in 3-5 mm in size were taken in 500 ml flask with 200 ml of water. This flask was connected to Clevenger type apparatus through special connector. A temperature was kept at 120 oC. Hydro distillation was performed from 3 hours. Oil obtained was dehydrated with sodium sulphate and then kept in air tight bottle in freezer. Hydro distillation setup is shown in figure 1.

For Microwave assisted distillation modification to Whirlpool VIP 34 microwave oven was done. A hole was made at top of the oven to hold a special connector that is then connected with condenser. Oven has a wave frequency of 2.45 GHz. It can deliver a maximum power of 1100 Watts. Flat bottom flask was used with 500 ml capacity. The setup is shown in figure 2. For microwave assisted hydro distillation (MAHD) the time range was 45 to 75 minutes with 15-minute interval and the ratio of peels to water was 1:2 (w:v). While for Solvent free microwave extraction (SFME) dry peels were subjected for extraction with a time range of 40 to 60 minutes with 10-minute interval. Energy calculations were done through digital clamp meter by measuring the drawing current.

III. RESULTS AND DISCUSSIONS

Experimental results for MAHD show that a yield of 1.2%, 1.7% and 1.8% was observed at 45 to 75 minutes time range with 15-minute interval respectively. A total increase of 34% was observed from initial reading, at 60 minutes while 6% increment occurs in yield from 60 to 75-minute time. Power consumption per gram was 0.42, 0.44 and 0.51 kWh/g for 45-, 60- and 75-minutes time. The increase in power consumption is very high for 75-minutes time i.e. about 16% as shown in table 1.

SFME results observed a yield of 1.4%, 1.7% and ≈1.7% for 40-, 50- and 60-minutes time with a yield increment of 22% and 4.2% for 50- and 60-minutes respectively. Power consumed per gram for 50-minute was 0.38 kWh/g with 11% increase while for 60-minute an increase of 10% i.e. 0.04 kWh/g was observed. For hydro distillation a experiment was conducted for 3-hours until a yield of 1.7% was obtained. Power consumed per gram of essential oil was the higher of all which is 1.3 kWh/g. As presented in table 2.
The aim of this research was to investigate the effectiveness of MAD. It was observed that MAD provides higher yields with less extraction time and low power consumption per gram of essential oil. Yield with connection to time and power consumption for SFME per gram was slightly and significantly better than MAHD and hydro distillation respectively. As shown in figure 3(a) and (b).

**Table 1. Results for Microwave Assisted Hydro Distillation in Comparison to Hydro Distillation**

| Parameter/Sample | 1 | 2 | 3 | Hydro Distillation |
|------------------|---|---|---|-------------------|
| Time (min)       | 45| 60| 75| 180               |
| % Yield (% Increased) | 1.2| 1.7 (34%)| 1.8 (6%)| 1.7 |
| kWh/g (% Increased) | 0.42| 0.44 (5%)| 0.51 (16%)| 1.3 |

**Table 2. Results of Solvent Free Microwave Extraction in Comparison with Hydro Distillation**

| Parameter/Sample | 1 | 2 | 3 | Hydro Distillation |
|------------------|---|---|---|-------------------|
| Time (min)       | 40| 50| 60| 180               |
| % Yield (% Increased) | 1.4| 1.7 (22%)| 1.8 (4.2%)| 1.7 |
| kWh/g (% Increased) | 0.34| 0.38 (11%)| 0.42 (10%)| 1.3 |

**CONCLUSIONS**

The aim of this research was to investigate the effectiveness of MAD. It was observed that MAD provides higher yields with less extraction time and low power consumption per gram of essential oil. Yield with connection to time and power consumption for SFME per gram was slightly and significantly better than MAHD and hydro distillation respectively. As shown in figure 3(a) and (b).

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