STUDIES ON REDUCTION OF EXTRACTION TIME OF SUPERCRITICAL CO_2 TO SAVE ENERGY

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Abstract

The present research work was conducted to examine the effect of changing geometric pattern of the extractor bed on the performance of supercritical co_2 extraction ($scco_2e$) of clove buds. The results of this study indicate that this modification of extractor bed geometry helps to complete this extraction process with higher yield in lesser time than conventional type of extractor bed. Since operating cost of extraction by supercritical fluid is higher than other methods, reduction of extraction time with better quality product in larger quantity significantly saves energy. $Scco_2e$ is already established as a promising novel technology for green extraction of natural products. Lot of research works are going on worldwide to reduce its energy consumption, thus cost of extraction to meet the challenges of the modern industrial development. This article explores a new research angle in the field of developing supercritical fluid extraction as an efficient, energy-saving, eco-friendly, sustainable technology for extraction of natural products from different plant materials.

Keywords: *Energy Saving, Green Extraction, Supercritical Co_2 Extraction, Extractor Bed Geometry.*

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1. INTRODUCTION

The challenges of the industrial development for green extraction of natural products like essential oils are multiple: more compact production units with reduced number of unit operations, developing innovative technologies with energy saving, cleaner and safer extraction protocols, limiting waste production, etc. Microwave-assisted extraction, ultrasound-assisted extraction,and supercritical fluid extractions (SCFE) are some of the novel techniques emerged in recent years for green extraction of natural products with a similar or better quality to that of traditional methods while reducing operational units thus helps to reduce total extraction time and save energy, minimize CO_2 emission and produce less waste [1,2]. From the point of reduction of stages, a single-stage process would appear to be ideal. The process of extraction by supercritical CO_2 ($SCCO_2E$) has this advantage. It uses no/very less amount of organic solvent(as co-solvent to extract polar component) and the time of extraction is very less than traditional extraction processes (like solvent extraction, distillation etc.) due to high selectivity, gas like diffusivity, liquid like solubility, high penetration rate of CO_2 in supercritical state [3,4]. Extract is completely solvent free as CO_2 is gas in atmospheric condition [4]. Use of CO_2 as solvent does not cause any additional green house effect as CO_2 is already produced in different processes, during respiration of animals and plants and present in the atmosphere. In conventional methods though extraction cost is less, most energy consumption occurred to recover the extract by evaporating the solvent and to eliminate residual solvent from the plant residue. Whereas in $SCCO_2E$ though extraction cost is higher but there is no solvent recovery energy cost. Yield and efficiency are also high in $SCCO_2E$ than above mentioned methods [3]. To reduce energy consumption technological modification may be possible in $SCCO_2E$ to recover the calories from conversion of CO_2 gas to liquid form and feed it into the heating system in the passage from liquid form to supercritical state. The efficiency of extraction methods need to be considered along with the cost of energy. This efficiency is based on the highest recovery of the effective constituents, the shortest processing time, use of minimum organic solvent, handling less amount of environmentally safe solvent. In this regard if the all steps of processes are taken into account, and not the individual extraction step, the $SCCO_2E$ has also emerged as an energy-saving technology.

In the present work $SCCO_2E$ of clove bud essential oil was conducted changing extractor bed geometry from the conventional packed bed geometry and its effect on extraction time and extraction yield were studied. Clove(scientific name *Syzygium aromaticum*) is one of the most important medicinal herbs and its bioactive components mainly eugenol, eugenol acetate, and β-caryophyllene possess antioxidant, antimicrobial, antifungal, anti-inflammatory, antiseptic, pain reliving and stimulating properties [8]. It is widely used in food, pharmaceutical, perfumery industries and aromatherapy. Comparative studies on clove oil extraction by different traditional methods and $SCCO_2E$ method established $SCCO_2E$ as the optimum extraction process for high quantity and high quality yield in shortest time [3]. To prevent channeling flow, to reduce dead space and for uniform distribution of the solvent use of glass beads along with solid sample was mentioned in literature [5,6]. The effect of length to diameter ratio of the extractor bed on SCF extraction was also provided in some literature [7]. But no previous references in literature are available where extractor bed geometry is directly changed to study its effect on essential oil extraction. In the present article the comparisons have been performed in terms of the total yield of clove oil and extraction time for three different types of bed geometry. Results show that this modification of bed geometry decrease extraction time significantly and increase oil yield in case of a particular modified bed condition. Reduction of extraction time means reduction of energy cost. Thus this study is a promising step towards the supercritical fluid extractor design to meet the challenges of efficient energy saving technology.

2. MATERIALS AND METHODS

2.1 Materials

Dried clove buds were purchased from local market of Haldia, West Bengal, India. All clove samples were stored in room temperature protecting from sunlight in closed plastic container to prevent any loss of volatile matters before further processing. The solvent carbon dioxide of above 99% purity was obtained from Bharat Oxytech Pvt. Ltd.; Haldia, West Bengal, India.

2.2 Preparation Of Solid Feed Sample

Dried clove buds were ground by a grinder to increase surface area as well as to rupture the cell wall before every run and the particle size was measured by mechanical sieving after extraction. An average particle size of 0.70±0.05mm was obtained for most of the experiments.

2.3 Experimental Apparatus

Experimental set up for SCFE was supplied by Chemtron Science Laboratories Pvt. Ltd., Mahape, Navi Mumbai, India. Its main components include one positive displacement pump to pressurize solvent CO_2 to required operating pressure, SCF generation unit, two parallel connected high pressure Extractors (each of 50mm inside diameter, 500mm height), one pressure reduction valve, and two low pressure Separators connected in series to confirm complete separation of extract from solvent. Bed geometry was changed by inserting a cylindrical channel coaxially with the conventional cylindrical extractor bed. Two different diameter channels were used to check their effect on extraction separately along with the conventional packed bed.

2.4 Supercritical CO2 Extraction

Details about supercritical fluid extraction methods are available in different literatures and books. For each of the preset extraction test only one extractor among two was packed with dried, ground clove buds under three different conditions of bed geometric. Experiments were carried out for varying condition of temperature (315K and 318K) keeping other parameters like operating pressure (190 ± 10 kg/cm^2), CO_2 flow rate almost constant for all these three types of bed geometry. CO_2 after solvent conditioning was pressurized through a high-pressure pump to the required operating critical pressure and entered in the SCF generator to raise the temperature to a desired value. $SCCO_2$ was then allowed to enter the extractor charged with solid sample. The extract-laden CO_2 was sent to the separators via the pressure reduction valve to precipitate out the extract completely into the separators. CO_2 gas after releasing extract is recycled back for further use until extraction is completed. The oil weight was measured periodically until extraction is completed.

2.5 GC/MS analysis of Clove Oil

GC/MS analyses were performed in the laboratory of Chemical Engineering Department, IITG, Assam, India.

3. RESULTS AND DISCUSSIONS

Main objective of this work was to study the effect of introduction of coaxial channel inside the extractor bed on total extraction time and total oil yield to verify whether this new invention can reduce extraction time significantly to save energy without any compromise with the quality of extract. For all these experiments pressure, particle size and CO_2 flow rate were kept constant. Table-1 shows extraction time and % yield for varying operating conditions of temperature, type of bed geometry, weight of feed for each of the SCFE runs. Fig. 1 and Fig. 2 show the effect of modification of bed geometry on extraction time and total yield content at two different temperatures. It can be noticed from the results that maximum yield was obtained when extractor was operated at 318K temperature using larger diameter channel (15mm) inside the conventional packed bed. Extraction time was also reduced /saved by almost 36 minutes for this run. The increase of temperature results in increase of oil yield in all types of bed geometry. Use of annular channel without any glass beads for extraction of clove oil increases oil yield for both types of modified bed when compared with normal packed bed. As per the results of GC/MS analysis the main components of clove oil identified were eugenol (58%), β-caryophyllene(11%) and eugenol acetate(22%) which are in good agreement with some of the articles. The main cause behind the less time requirement for extraction along with better quality, large quantity yield content by this modified bed geometry is significant reduction of the resistance to mass transfer process which enhances contact between both phases thus increasing extraction rate significantly.

Table - 1: Experimental Results for Extraction of Clove Oil with SCCO2

Run No.	Extractor Bed type	Operating Temp (K)	Sample Weight (gm)	Extraction Time (min)	% Yield (gm oil /gm solid Feed)*100
1	Normal Packed Bed	318	450	203	11.00
2	Extractor Bed with Large Dia. Channel	318	450	167	16.40
3	Extractor Bed with Small Dia. Channel	318	450	199	15.00
4	Normal Packed Bed	313	250	99	8.60
5	Extractor Bed with Large Dia. Channel	313	250	110	13.35
6	Extractor Bed with Smalll Dia. Channel	313	250	163	11.00

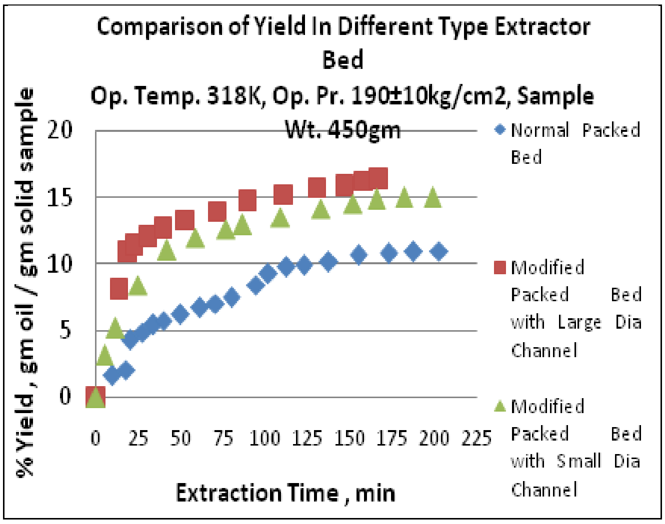

Fig-1: Effect of Extractor Bed Geometry on Extraction Time and yield Content

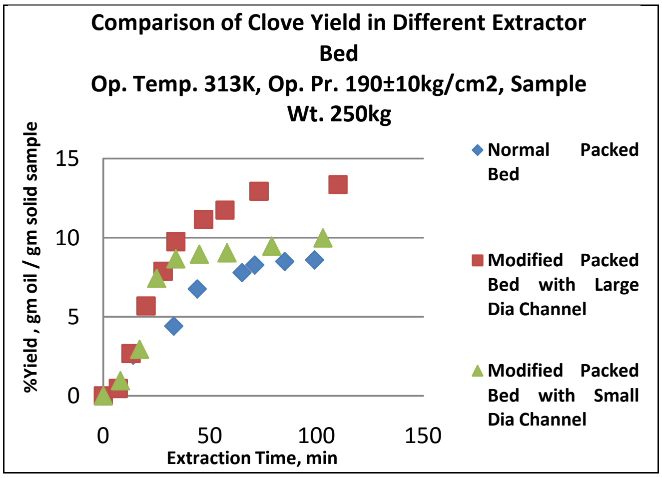

Fig-2: Effect of Extractor Bed Geometry on Extraction Time and yield Content

CONCLUSION

A modified static bed with varying annulus space was manufactured in this work to measure the yield of clove extract using supercritical CO_2. The obtained yields with respect to time clearly indicate its edge over conventional fixed bed. Reduced extraction time with increased oil yield was indication of efficient extraction in modified bed compared to normal bed. The incorporation of perforated hollow cylinder in conventional fixed bed brings differences for the leached solids on accessibility, percolation and diffusion by the SCF. These factors can have a strong effect on improved yield and cost reduction.

ACKNOWLEDGEMENTS

This work was partly supported by AICTE under MODROB scheme to develop the laboratory for supercritical extraction. This work was financially supported by IIChE, Kolkata, India through project titled “Supercritical Fluid Extraction of Natural Products from Various Plant Resources”.

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