STUDY OF ESSENTIAL OIL DISTILLATION UNIT - CASE STUDY FOR MENTHA CROP

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by

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Dedicated to My Parents

and my beloved Nisheet

Certificate

This is to certify that the thesis entitled **"STUDY OF ESSENTIAL OIL DISTILLATION UNIT - CASE STUDY FOR MENTHA CROP"** being submitted by **Mr. Rajesh Katiyar** for the award of the degree of DOCTOR OF PHILOSOPHY is a record of the original bonafide research work carried out by him under my guidance and supervision at the Centre for Rural Development and Technology, Indian Institute of Technology, Delhi. The work presented in this thesis has not been submitted elsewhere either in part or full to any other University or Institute for award of any degree or diploma.

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New Delhi Rajesh Katiyar

Abstract

Oil extracted from *Mentha arvensis* L., popularly known as corn mint, having menthol as a major component is widely used in flavor, fragrance and pharmaceutical industry. Crop is aromatic member of the Lamiaceae family whose essential oil is the richest source of natural menthol. Around 225 x 10^3 ha of land gets cultivated in the mint belt in geographical area of Indo-Gangetic plains. The share of India in world mint oil market is around 55% to 60% and produces around 35,000 metric ton of mint oil annually. Study of recovery of *Mentha arvensis* L. oil in field level distillation units was done and process at laboratory level was studied. Both fresh as well as partially dry crop was utilized for oil extraction. Process material balance identified the loss routes of the essential oil during extraction. GC and GCMS analysis was done on the capillary column under temperature programmed condition, and total 30 components were identified. Laboratory, pilot plant level studies and mathematical modeling of kinetics of oil recovery helped in identifying drawbacks of field level units. Possible utilization of by-products of the process was explored, as 80% of the units used by small and medium farmers are water-steam distillation type. This sector would be benefited by the results of the study carried out.

Water-steam distillation process is used extensively for essential oil extraction, and yield through this process becomes important as oil is present in plant in low concentrations. Field level studies provided information on the agricultural practices, process and operational details of the units leading to identification of areas in which improvements were possible. Preliminary laboratory distillation set-up similar to field unit, was utilized for extraction of oil. Clevenger set-up was utilized at various stages to obtain oil content of the crop. Essential oil content determined by

Clevenger method under standard conditions for fresh crop was 0.83 ± 0.01%. Yields obtained with water-steam distillation preliminary laboratory set-up was evaluated for its performance with the help of the results obtained from standard procedure of Clevenger run for the crop of the same lot. Process mass balance and composition analysis confirmed that oil which is not getting extracted and remains with the crop is rich in menthol.

The main objective of the laboratory water-steam distillation modified set-up was to extract oil from crop to a maximum value, reaching as near as oil content in the crop. In modified set-up, around 1 g more oil was obtained from 1 kg of fresh crop in comparison to preliminary set-up. Material balance results established that losses through various routes were reduced. Oil that was extracted from modified set-up from fresh crop, contained menthol (77.01%) in good amount and other major valuable components like menthone (5.15%), isomenthone (3.12%), menthyl acetate (4.55%), neomenthol (2.63%) and isomenthol (0.32%) were in acceptable range along with the other components qualitatively. Kinetic studies of the oil yield from fresh crop was carried out keeping crop mass, feed water for steam generation, pack density of the crop constant, and steam rate was varied. The amount of distillate water collected provided idea of the steam utilized for the process. With the increase in steam rate, cumulative oil yield decreased. Study on kinetics of oil yield from the partially dry crop revealed, that for nearly same steam flow rate, partially dry crop provides around 15% more yield in comparison to fresh crop. For partially dry crop, loss in drying was around 24%, cumulative oil yield was 0.795% and steam rate was 0.75 ml/min. On the basis of drying studies and observations as well as results of Clevenger runs, and oil yield obtained from modified set up, it was established that drying range from 20% to 30% weight loss should be preferred. In the oil extracted from fresh crop, menthol was 77.01% and in the oil extracted from partially dry crop, menthol was 77.84%. As regards to the remaining components, the difference was in very slight variation.

For the purpose of extension and verification of the laboratory level results that were obtained, runs on pilot scale water-steam distillation unit were carried out. The unit was also utilized for obtaining yield data and parameters to scale up to field level, and oil extraction was carried out with partially dried crop as well as for fresh crop. Maximum oil extracted was 7.75 g per kg of the crop for partially dried crop to the extent of 27.8% loss in weight. In fresh crop, oil extracted was 6.96 g per kg of crop. At laboratory level, maximum oil extracted was 7.95 g per kg of crop for partially dry crop whereas for fresh crop it was 6.72 g. The shade dried crop in all cases has provided higher yield than the maximum yield obtained from fresh crop. Composition of major components, β-pinene (0.76%), β-myrcene (0.61%), limonene (2.32%), menthone (5.06%), isomenthone (3.63%), menthyl acetate (3.37%), neomenthol (2.90%) and menthol (77.58%) of oil extracted from partially dry crop confirm to its good quality and meet the specifications of IS 528 : 1999, that is, Oil of *Mentha Arvensis* – Specification. Oil has a very good percentage of menthol content above 77%, meeting high commercial value.

The initial distribution of solute within the solid substrate influences selection of possible model of essential oil extraction from a crop. Mint species have major portion of oil born on its surface and essential oil storage in glandular trichomes on leaf surface is a feature of the family Lamiaceae. To model the yield of Mentha arvensis L. essential oil extraction process from its crop, model based on one-dimensional unsteady-state diffusion through flake particle (flat geometry) was utilized. Experimental yield data on laboratory modified set up and pilot scale unit were used for validation of the model. Diffusivity, D value obtained for the water-steam distillation pilot scale run was 1.02877×10^{-11} m^2/s. Experimental results of *Mentha arvensis* L. oil extraction yield agree very well with the flake particle based unsteady state diffusion model.

Study of different characteristics of the spent biomass obtained after oil extraction was carried out for comparison with different agricultural residues to explore its feasibility in better use as alternative renewable bio-fuel. Physical and chemical characteristics of spent biomass were examined for calorific value, ash content, TGA and CHNS. Distillate water being other important by-product of process was examined, for its oil content. Oil in distillate water was recovered through solvent hexane. The composition analysis of oil recovered from distillate water established that menthol (81.06%) was present in higher percentage in comparison to oil that was extracted from fresh crop as well as from partially dry crop. For efficient runs, oil in distillate water was on lower side. On basis of the study results and recommendations made regarding minor modifications and better operation, an efficient operating procedure under standard conditions of operating parameters, for an existing water-steam distillation field unit has been suggested. This would result in around 50% water saving, 30% increase in oil yield, minimum 1 hour saving in batch time and around 15% to 20% saving in fuel.

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