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Optimization of factors influencing Solvent Extraction of Frankincense using Taguchi's Method

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ABSTRACT

Solvent extraction technique was employed for the extraction of chemical constituents of the frankincense from Boswellia serrata. The effects of feed size, time of contact and the type of solvent on the extraction yield were investigated using Taguchi's design of experiments method. The data were analyzed using ANOVA and the experimental results showed that hexane was a better solvent than ethyl acetate and methanol. An optimal setting of feed size (0.5 mm), contact time (3 h) and hexane as solvent was obtained under which the yield was 98.065%. Through this investigation, the Taguchi's approach was successfully applied and validated. Based on the quality of the yield, extraction appeared to be a more efficient route than steam distillation for obtaining the pharmaceutically significant ingredients of frankincense from Boswellia serrata.

Keywords: Boswellia, Taguchi design, Solvent extraction, Optimization.

1. INTRODUCTION

Frankincense, or olibanum, is the oleogum resin that is harvested from several different trees belonging to the genus *Boswellia*. Because of its noteworthy scent and use as an important fixative in perfumes, soaps, creams, lotions, and detergents, the perfume and cosmetic industry has considerable interest in the production of frankincense. In addition, since ancient times, it has been also used in folk medicine for its antiseptic, antiarthritic, and anti-inflammatory effects due to which many pharmaceutical companies are having growing interest in extraction and isolation of chemical constituents products from frankincense. For these reasons, since the last decade, significant research leading to defining the medical effects of frankincense and identifying constituents that are responsible for these effects is ongoing. Frankincense is found in Ethiopia, Somalia, Yemen, Oman, India and Pakistan.

The oleo gum-resins contain 30-60% resin, 5-10% essential oils, which are soluble in the organic solvents, and the rest is made up of poly-saccharides. Extraction methods can be time consuming, require the use of large amount of organic solvent and may have lower extraction efficiencies (Ong, 2004). Moreover, even with the same technique of extraction, for different constituent compounds in different plant species, different operating conditions may be required (Noriega et al., 2012). Thereby, the method and processing conditions employed in the extraction of chemical compounds from gum resin as raw materials play important roles in determining the quality, cost and, overall, the efficacy of the standardized pharmaceutically important intermediate product (List &Shmidt, 1989). Based on these considerations, it is of great interest to undertake studies in order to investigate the relationship between extraction parameters and extraction yield.

The aim of this work was to study the effect of feed size, time of contact and the type of solvent on the extraction yield and determining the optimum set of conditions for the extraction using Taguchi's design of experiments method.

2. LITERATURE REVIEW

Earlier research investigations have shown that oleo gum resin of *Boswellia serrata* to have anti-inflammatory, anti-carcinogenic and anti-tumour properties. The anti-inflammatory activity is mainly attributed to the presence of major constituent of pentacyclic triterpene namely α-boswellic acids and β-boswellic acids (Abdel-Tawab et al., 2011). Amongst these constituents 3-acetyl-11-keto-β-boswellic acid is regardred the most active and potent anti-inflammatory agent and also has medicinal properties against cancer, arthritis, bronchial asthma, crohns disease and chronic colitis (Gerhardt et al.,2001). Culioli et al., (2003) extracted and purified acetylated boswellic acid using methanolic extract followed by silica gel column chromatography. Faruck et al., 2006 developed a protocol to extract heavy terpenes from frankinsence (*Boswellia sacra*) by simple soxhlet extraction method. Their chemical profiling data revealed that α-pinene (61.56%) was the major component of this heavy oil followed by α-amyrin (20.68%) and β-amyrin (8.18%).

This data was in accordance with earlier reports on frankinsence essential oil extracted by hydro distillation method, where α-pinene is the principle component (Baser,2003). Furthermore it was found that frankinsence from *Boswellia sacra* restricted the growth of pathogens like *Klebseila*, *E.coli*, *staphylococcus* and *bacillus*. Niphadkar and Rathod, 2017 concluded that application of ultrasound assisted extraction resulted in intensification of extraction yield of 3-acetyl-11-keto-β-boswellic acid from *Boswellia serrata*. The main mechanism for intensification of extraction with ultrasound assisted extraction was cavitation phenomena, which caused disruption of plant tissue and resulted in enhanced mass transfer. Process parameters such as extraction time, solute to solvent ratio, duty cycle, temperature, ultrasound power, and ethanol concentration had significant influence on ultrasound assisted extraction. Ultrasound assisted extraction compared with conventional extraction process such as soxhlet and batch extraction required lesser extraction time.

This study indicated that ultrasound assisted extraction was a more promising method for process intensification with advantages like reduced operation time of extraction and enhanced recovery of 3-acetyl-11-keto-β-boswellic acid from *Boswellia serrata*. The results established by Niphadkar and Rathod, 2017 using the Box-benhken method indicated that the interaction between pressure and extraction time was found to be more significant process parameters for enhancing the yields of 3-acetyl-11-keto-β-boswellic acid. Yuan et al (2008) used acid base precipitation method followed by column chromatography purification for isolation and purification of 3-acetyl-11-keto-β-boswellic acid. Lin et al., 2013 extracted biologically active compounds from *Boswellia sacra* gum resins using hydrodistillation and investigated their benefits in anti-cancer therapy. They reported that in order to prepare frankincense essential oil for anti-cancer therapy standardized procedures to harvest, store and process *Boswellia* species gum resins have to be established to obtain consistent results. Although literature reports available on the medicinal benefits of extracts from frankincense are abundant very little information is available on the process parameters desirable for the extraction of boswellic acids from frankincense. Therefore in the present study an attempt has been made to study the most significant parameters and their influence on the extraction yield. Through this study the optimization of the process parameters using experimental design technique has been demonstrated.

3. MATERIALS AND METHODS

Frankincense was purchased from the departmental store of Kerala Ecotourism centre of Konni and crushed and particle subjected to sieve analysis. Frankincense extraction was carried out using Soxhlet extraction apparatus using laboratory reagent grade solvents purchased from the local market. The process parameters and their levels used for the extraction are depicted in Table 3.1.

Table 3.1 Influencing Factors and their levels

Factor	Level 1	Level 2	Level 3
Particle Size (mm)	0.5	1.5	2.5
Contact Time (h)	1.0	2.0	3.0
Solvent	Hexane	Methanol	Ethyl Acetate

In order to determine the optimum conditions during solvent extraction experiments, design of experiments by Taguchi method was used. The factors and their levels selected to determine the optimum solvent extraction conditions, using Taguchi orthogonal array (L9) are shown in Table 3.2.

Table 3.2. Taguchi's L9 Orthogonal Array for the Extraction Process

Experiment No.	Particle Size (mm)	Contact Time (h)	Solvent
1	0.5	1	Hexane
2	0.5	2	Methanol
3	0.5	3	Ethyl Acetate
4	1.5	2	Hexane
5	1.5	3	Methanol
6	1.5	1	Ethyl Acetate
7	2.5	3	Hexane
8	2.5	1	Methanol
9	2.5	2	Ethyl Acetate

4. RESULTS AND DISCUSSION

4.1 Solvent Extraction Process

In this study, the particle size and contact time were selected as the influencing factors when the extraction was investigated with three different solvents. All the experiments are presented in the Taguchi orthogonal array for the solvent extraction process, and the results of this process are shown in Table 4.1.

Table 4.1. Taguchi's Design of Experiments with extraction yield

Experiment No.	Particle Size	Nature of Solvent	Contact Time	Extraction Yield (%)
1	0.5	Hexane	1	92.66
2	0.5	Methanol	2	11.85
3	0.5	Ethyl Acetate	3	78.55
4	1.5	Hexane	2	74.92
5	1.5	Methanol	3	09.95
6	1.5	Ethyl Acetate	1	60.05
7	2.5	Hexane	3	96.67
8	2.5	Methanol	1	07.55
9	2.5	Ethyl Acetate	2	74.93

4.2 Main Effects Plot

After performing the experiments as per Taguchi's experimental design, main effects plots for maximum extraction yield are plotted for the significant factors and their levels. A main effect is a direct effect on parameters on response and dependent variables. Typical main effect plots of parameters with respect to the yield from the extraction are shown in Fig 1-3.

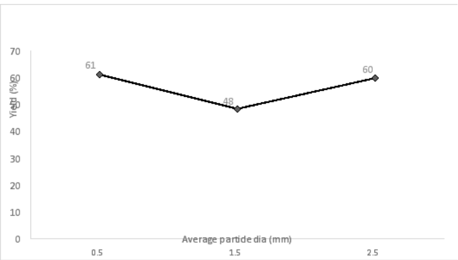

Fig 4.1. Effect of Particle size

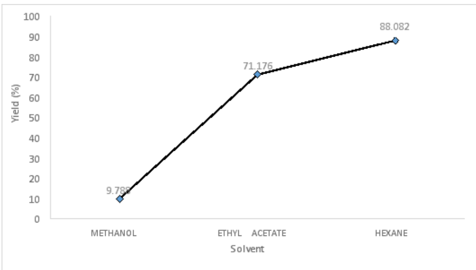

Fig 4.2. Effect of type of solvent

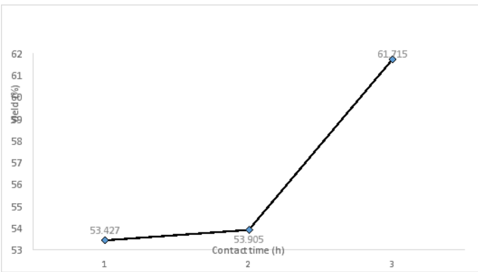

Fig 4.3. Effect of contact time on yield

Based on the criteria, the higher the best for the extraction process, the optimum process parameters were predicted as 0.5mm particle size with hexane as the extraction solvent for three hour contact time. The yield based on the predicted parameter combination was found by experimental validation to be 98.065 %.

4.3 Analysis of Variance

Collected date from Taguchi design of experiment was analyzed using ANOVA method. ANOVA method accounts for the variables from all involved sources including error term. The relative contributions of the factors were determined by comparing their variances. The analysis of variance for the data is given table 4.2

Table 4.2. Analysis of variance (ANOVA)

Factors	DOF	Sum of squares	Variance	Error (%)
Particle size	2	294.063	147.03	2.76
Type of solvent	2	10183.92	5091.96	95.59
Contact time	2	139.543	69.77	1.31
Error	2	35.28	17.64	0.331

5. CONCLUSION

In this study, the optimum conditions for solvent extraction process of frankincense were found to be when using particle size (0.5mm), contact time (3h) and hexane solvent, which were determined by Taguchi method. From this investigation, it was shown that parametric optimization and factors influencing the extraction process can be well predicted. Moreover, there is substantial saving of cost and time in experimental work through this experimental design approach. The results obtained during this laboratory study showed the possibility of extracting valuable chemical constituents from Indian frankincense and that further research can be conducted to find the other practical conditions to utilize this raw material to produce and provide an economical source of frankincense in India.

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