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# Comparative Study of Microwave Assisted With Conventional Extraction of Calcium Sennosides from Senna Leaf

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**ABSTRACT**

A calcium sennoside was extracted by developed microwave assisted extraction technique from Senna leaves. Microwave extraction was carried out at 250 W for 20 minutes, 350 W for 15 minutes and 450 W for 10 min heating period, these produced increase yields of calcium sennosides when compared to conventional heating. The yield of calcium sennosides obtained by microwave extraction was found to be 3.0 gm, 3.4 gm and 3.9 gm respectively, more than that obtained by conventional method .The microwave assisted extraction method reduced the amount of solvent consumed for the extraction purpose and also reduced the time of extraction. This was also aided due to the increased intensity of microwave power. The calcium sennoside products obtained by Microwave and conventional methods were same both physically and chemically. The method developed can be used mainly for routine laboratory scale isolation of calcium sennosides from Senna leaves.

**Keywords**: - Microwave extraction, calcium sennosides, Senna, intensity, Methanolic extract.

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## INTRODUCTION

Plants synthesize a most number of secondary metabolites which are important from therapeutic point of view. Extraction of crude drugs for these secondary metabolites can be done by various processes depending on the physical nature of the drug and chemical properties of the constituents present in it. Various traditional methods used for the extraction of drugs include Infusion, Decoction, Digestion, Maceration and Percolation. Out of these Maceration and Percolation are of particular importance and most Pharmacopoeias refer to these processes for the extraction of crude drugs. The conventional extraction processes are time consuming, e.g., maceration done for 2-7 days; involve bulk amount of solvents and ultimately there might be thermal decomposition of the target molecule as in the case of Soxhlet extraction.

The demand for new extraction techniques has encouraged the development of alternative extraction techniques such as Ultrasonic Assisted Extraction (UAE), Microwave Assisted Extraction (MAE), Supercritical Fluid Extraction (SCF) and Accelerated Solvent Extraction (ASE) [1]. These techniques have enabled automation, shortened extraction time and reduced organic solvent consumption.

Microwave-assisted extraction (MAE) is a relatively new extraction technique, which utilizes microwave energy to heat the solvent and the sample and to increase the mass transfer rate of the solutes from the sample matrix into the solvent. The usage of microwaves for extracting plant constituents is still in infancy. The microwave-assisted extraction (MAE) technique is a promising technique which is highlighted by increased extraction yield high quality products in decreased time and solvent consumption; moreover the reproducibility is better [2].

Microwave-assisted extraction is a process of applying the microwave energy to a liquid-solid system and partition compounds of interest from the solid sample into the surrounding solvents. The special heating mechanism of microwave and the fact that different chemical substances absorb microwave to different levels make microwave-assisted extraction an efficient method for extraction and more importantly make selective extraction of target compounds possible. The microwave-matter interaction is mainly caused by the interaction of the microwave with the polar molecules. In a polar molecule, due to the difference of the electro negativity of different atoms and the specific structure of the molecule, the whole molecule exhibits a partial positive charge and a partial negative charge and forms a dipole [4].
The microwave-assisted extraction process is a combination of different effects. When a polar solvent with a relatively high dielectric constant and loss factor is used the solvent will be heated by the microwave energy through dipole rotation. From the above description it is quite understandable that microwave assisted extraction is a very efficient method for extraction of key components from plant materials.

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## MATERIALS AND METHODS

**Conventional Extraction of Calcium Sennosides from Senna leaves [3]**


## PROCEDURE

20g of powdered senna leaves was extracted with 75ml benzene for 15min on electric shaker, filtered in vacuum and solvent distilled off. The left over marc was dried at room temperature and extracted with 75ml of 70% methanol for 25min on electric shaker, filtered under vacuum. The marc was re-extracted with 50ml of 70% methanol for 15min, filtered and the methanolic extracts combined. The methanolic extract was concentrated to 1/8th volume, acidified to pH 3.2 by adding $HNO_3$ with constant stirring. It was set aside for 15min at 5°C, filtered and 1g of anhydrous calcium chloride in 12ml of denatured spirit was added with constant stirring. The pH of the solution was adjusted to 8 by addition of potassium hydroxide (KOH) and set aside for 15min. above these procedures were study in three cases at different time for extraction on electric shaker. The precipitate obtained in all the three cases were collected, dried and weighed. The percentage yield was also calculated.

**Conventional extraction product 1**

**Conventional extraction product 2**

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**Conventional extraction Product 3**

**Microwave Extraction of Calcium Sennosides from Senna leaves**

Calcium Sennoside from senna leaves was extracted by varying the parameters of amount of solvent used, temperature and time. The extraction was performed thrice with varying results.

**MICROWAVE EXTRACTION OF CALCIUM SENNOSIDES**

**Procedure**

20g of powdered Senna leaflets were extracted with varying amounts of benzene for different time intervals and varied power of Microwave thrice, filtered in vacuum and the solvent distilled off. The left over marc was dried at room temperature and again extracted with varying amounts of 70% methanol for different time intervals and varied power of Microwave thrice.

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MAE 1 Product

MAE 2 Product

MAE 3 Product

The marc was re-extracted with varying amounts of 70% methanol for different time intervals and varied power of Microwave thrice, filtered and the methanolic extracts combined. The methanolic extracts were concentrated to 1/8th volume, acidified to pH 3.2 by adding $HNO_3$ with constant stirring. The mixtures were set aside at different time intervals in all the three cases at 5°C, filtered and 1g of anhydrous calcium chloride in 12ml of denatured spirit was added with constant stirring. The pH of the solution was adjusted to 8 by addition of potassium hydroxide (KOH) and was set aside at different time intervals in all the three procedures. The precipitate obtained in all the three cases were collected, dried and weighed. The percentage yield was also calculated.

**General conformation tests for calcium sennosides**

**Borntager's Test**

To 3 ml sample, dilute sulphuric acid ($H_2SO_4$) was added, boiled and filtered. To the cold filtrate, equal volume of benzene was added and shaken. The organic solvent was separated and ammonia was added. It was observed that the ammoniacal layer turned pink confirming the presence of anthraquinone glycoside presence.

**Modified Borntager's Test**

To 5ml sample, 5ml Ferric chloride and 5ml dilute hydrochloric acid was added. It was then heated for 5min in boiling water bath. Cooled and benzene was added. It was shaken well, separated and equal volume of dilute ammonia was added. It was observed that the ammoniacal layer turned pink confirming the presence of anthraquinone glycoside.

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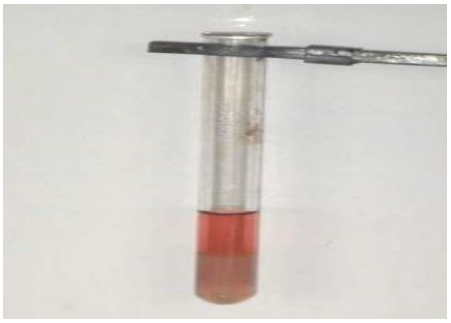
Borntager's Test

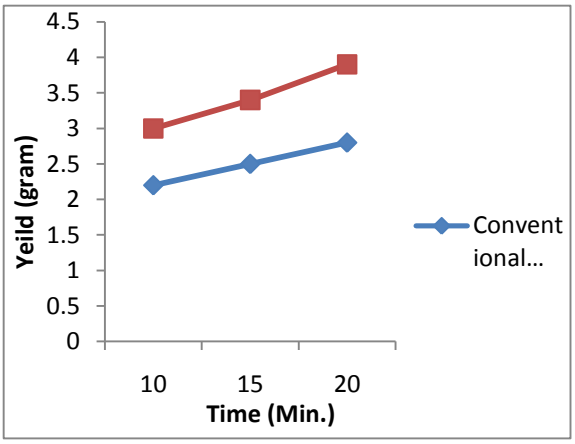


Modified Borntager's Test

## RESULTS AND DISCUSSION

| S.No. | Conventional Extraction | | | Microwave Extraction | | | |
|---|---|---|---|---|---|---|---|
| | Duration (m) | Yield (g) | Amount of Solvent used (ml) | Intensity (W) | Duration (m) | Yield (g) | Amount of Solvent used (ml) |
| 1 | 10 | 2.2 | 100 | 250 | 20 | 3.0 | 60 |
| 2 | 15 | 2.5 | 100 | 350 | 15 | 3.4 | 40 |
| 3 | 20 | 2.8 | 100 | 450 | 10 | 3.9 | 20 |

**Fig Graph: Comparision of Conventional Ectraction and Microwave Extraction Methods.**




## CONCLUSION

This study was to replace conventional extraction by the Microwave assisted extraction. Microwave assisted extraction (MAE) required less time, small quantity of solvents, higher extraction rate and better product with minimum loss. There is also less risk of decomposition and oxidation of Phytoconstituents from plant. It was also found that increase in the power of microwave increased the yield by 3 - 3.9 gm along with reduced solvent consumption and time. However, there was a limiting factor on the reduction in the quantity of solvent. The results

were found to be encouraging and therefore it is recommended that extraction of calcium sennosides from senna leaves be carried out by microwave at higher power using small amount of solvent and minimum time.

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