

## Isolation and Study of $\beta$ -sitosterol the Unsaponifiable Matter from the Plant *Malachra capitata* (Linn)

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### ABSTRACT

The fat obtained from the shade dried plant *Malachra capitata* (Linn) was saponified and the unsaponifiable matter was isolated. The unsaponifiable matter was identified as  $\beta$ -sitosterol by chemical and spectroscopic analysis.

**Key words:** *Malachra capitata* (Linn), N.O.- Malvaceae, unsaponifiable, matter,  $\beta$ -sitosterol.

### INTRODUCTION

The plant *Malachra capitata* Linn in commonly known as Ranbhendi in Bombay. It belongs to natural order Malvaceae.

The plant occurs in thought the hotter parts of India. It is used as emollient. experimental

About 500 gm the plant *Malachra capitata* (Linn) was shade dried, powdered and extracted with petroleum ether (40-60°) in a Soxhlet extract for about 70hrs.

The petroleum ether (40-60°) extract was concentrated to a get a yellow viscous fat.

About 50 gm of the above fat was saponified by refluxing it with alcoholic potassium hydroxide. The soap thus obtained was dissolved in water and the unsaponified matter was recovered by extraction with ether in a separating funnel.

#### Study of the unsaponifiable matter

The ether from this ethereal extract containing the unsaponifiable matter was distilled under reduced pressure to get the unsaponifiable

matter which was when dried over anhydrous sodium sulphate, gave a light yellow product.

On CH, analysis, the unsaponifiable matter analysed for molecular formula  $C_{29}H_{50}O$ ,  $M^+ = 414$ , m.p. 134-35°C and  $(\dots)_D^{22} = -36^0$  (in  $CHCl_3$ ).

The unsaponifiable matter also responded positively to following; colour reactions, which are characteristic of steroids.

- 1) A Yellow colour changing to red in Salkowski reaction<sup>2</sup>,
- 2) A violet red colour in Tschugajew reactions<sup>3</sup>,
- 3) Intense pink colour changing to violet in Noller's reactions<sup>4</sup> and
- 4) A red violet colour in Liebermann-Burchard reaction<sup>5</sup>.

The unsaponifiable matter showed the wave length of maximum absorbance at MeOH

$$\lambda_{\max} = 209\text{nm}$$

Significant bands were observed in the IR spectrum of the unsaponifiable matter and the structural units inferred with the help of available literature<sup>6-9</sup> were at; 3358 (-OH), 2908 (-CH<sub>3</sub> - CH<sub>2</sub>), 1640 (>C = CH stretching), 1452 (-CH<sub>3</sub>), 1370, 1135,

1070, 1018 (Triterpenoidal) 958,996 (Cyclohexane ring),  $850\text{ cm}^{-1}$  ( $>\text{C} = \text{CH}$  deforming).

KBr

The band at  $\lambda_{\text{max}}$  at  $1640\text{ cm}^{-1}$  in the IR spectrum showed the presence of unsaturation in the unsaponifiable matter which was further confirmed because it gave positive test with TNM.

Another band at  $3358\text{ cm}^{-1}$  indicated the presence of -OH group(s) in the compound. The number of -OH group(s) was determined by the acetylation of the unsaponifiable matter which were estimated by acetylation with  $\text{Ac}_2\text{O}$ /Pyridine to get an acetylated derivative, having molecular formula  $\text{C}_{31}\text{H}_{52}\text{O}_2$ ,  $M^+ = 456$  and (m.p.  $143\text{-}44^\circ\text{C}$ ). The percentage of acetyl group (10.64%) was estimated by the procedure of Wiesenberger<sup>10</sup> as described by Belcher and Godbert<sup>11</sup>, when it showed the presence of only one -OH group in it.

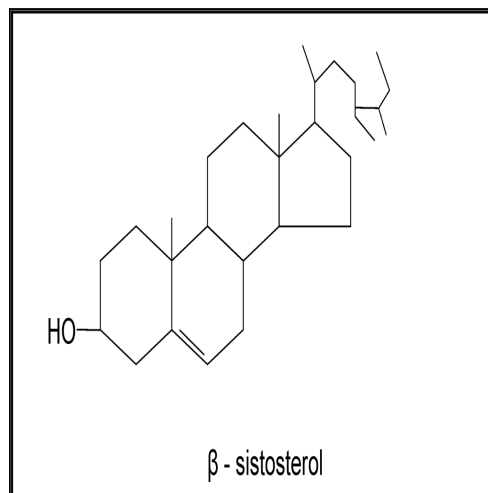
The formation of acetyl derivative was further supported by the appearance of the acetyl absorption band at  $1720\text{ cm}^{-1}$  in the IR spectrum of acetyl derivative and disappearance of the hydroxyl absorption in the IR spectrum of the acetylated unsaponifiable matter. The presence of only one acetoxy group (3H) was also confirmed by the signal at  $\delta = 2.03$  in the  $^1\text{H}$ -NMR<sup>12-13</sup> spectrum of the unsaponifiable compound.

The signal for the proton at  $\delta = 4.526$  further established the secondary nature of the hydroxyl group.

The unsaponifiable matter when oxidized with chromic acid yielded an oxidation product, m.p.  $133\text{-}34^\circ\text{C}$ , molecular formula  $\text{C}_{29}\text{H}_{48}\text{O}$ , and  $M^+ = 412$ , which was found to give positive Zimmermann test and confirmed the presence of 3-keto group. This observation concluded that the -OH group must be at C-3 and further confirmed that it must be secondary<sup>14</sup>.

## RESULTS AND DISCUSSION

The deep sweep in the available literature<sup>15-16</sup> concluded that the unsaponifiable matter under examination was identical with the known compound  $\beta$ -sitosterol (mmp, Co-PC, Co-TLC) (I). The identity of the compound was further confirmed by mmp. Co-TLC with authentic sample of  $\beta$ -sitosterol. Compilation of all above facts finally identified the unsaponifiable matter as:  $\beta$ -sitosterol.



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