

Isolation of Chemical Components from Rosa rubiginosa and Synthesis of 2'-Hydroxy Chalcone

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Abstract: The present work was carried out to investigate and separate the chemical components present in the Rosa rubiginosa and to perform a synthesis of 2'-Hydroxy Chalcone a natural product. Roses are widely distributed in various parts of the world. The chief component present in any of the species of Rose is essential rose oil. This rose oil consists of hundreds of components in it and separation of these components is quite difficult. The work was divided into two parts. In the first part, extraction of fresh petals of Rosa rubiginosa was carried out by solvent extraction method using hexane as a solvent and four components were isolated. In the second part, a synthesis of 2'-Hydroxy Chalcone was done. Literature study includes two methods for the synthesis of Chalcone. One is Claisen-Schmidt condensation and other is Aldol condensation. The method adopted to carry out the synthesis of 2'-Hydroxy Chalcone was Aldol condensation in the presence of sodium hydroxide because it yielded a good quantity of product. Different Chromatographic techniques like column chromatography and thin layer chromatography, as well as spectroscopic techniques like IR, UV, and NMR, were used for identification of the products.

Keywords: Extraction, Isolation, Identification, Rose oil, 2'-Hydroxy Chalcone

I. Introduction

Extraction of natural products or the chemical components present in plant species is one of the most challenging researches for chemical and pharmaceutical industries. Phenolic compounds are naturally found in plants. These phenolics compounds include anthocyanin, Carotenoids, flavonoids and are also found in legumes, vegetables, and herbs. They help to protect against free radical damage. Therefore the consumption of these antioxidants helps to protect the human body against diseases like diabetes, cancer, cardiovascular, etc. Among the natural compounds, essential oils are also used as food flavors, perfumes, and as raw materials for pharmaceutical industries. Rose essential oil possess health beneficial properties like anti-depressant, antiseptic, antispasmodic, antiviral, bactericidal, hepatic, laxative and uterine substance. Roses are considered as most beautiful flowers and are widely distributed. Many species of roses have been used for its medicinal values i.e. to cure menstruation problem, fight depression, treat wounds, prevents excessive bleeding. The genus Rosa L. consists of approximately 150 species. Rosa rubiginosa also known as sweet briar or eglantine or Rosa canina is a species of rose that is found in Europe, Northwest Africa, and western Asia. They are grown because the syrup obtained from this species contain three times of Vitamin-C than in blackberry. They are also used to make tea in the region where they are grown in abundance. They are taken as a tea to fight against viral infection and to treat urinary and kidney tract disorder. This rose species is the most abundant and most variable. They are dense, deciduous shrub up to 10 feet high. These flowers are valued for their fragrant

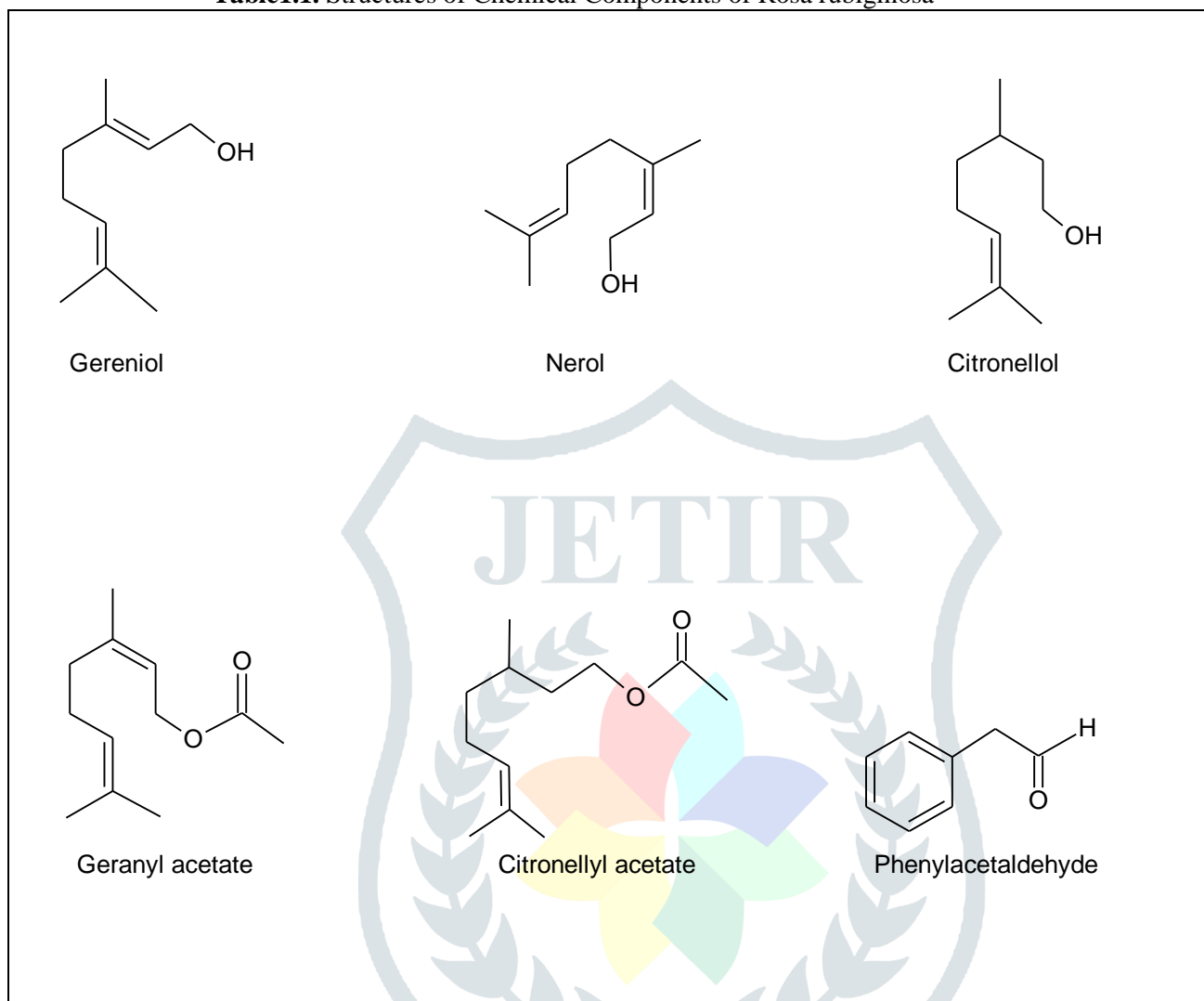
Their distinguishing features are as follows:

- They grow upright or sometimes as climbers.
- Their stems are smooth and contain prickles which are 10-15mm long.
- They possess compound leaves with 5-7 leaflets and sharply toothed margin.
- Flowers of these species are sweetly scented. They have red, white or pink petals.
- Fruit of these species is oval or egg-shaped that turns orange or red when it matures.
- Fruits sometimes have spines near their bases.

Essential oil is the main component found in the petals of all the roses and it takes a huge amount of rose petals to obtain a small amount of essential oil. There are two types of oil produced from the roses, rose absolute oil and rose otto oil. Rose absolute oil is obtained from solvent extraction. Rose Otto oil is extracted by gentle distillation and is mainly obtained from Damask rose. The essential oil obtained from the petals of Rosa rubiginosa consist of a mixture

of chemical components like geraniol, nerol, Citronellol, citric acids, tannins, flavonoids, isoquercitrin, invert sugar, lycopene, malic acid, pectin, sucrose, succinic acid, α -tocopherol.

Table 1.1. Structures of Chemical Components of *Rosa rubiginosa*



Many extraction methods are used for the extraction of chemical components from rose oil and are dependent on the type of plant material being used. Traditional techniques like solvent extraction and distillation take more time, provide fewer yields and use a large amount of organic solvent. New methods have been developed like Microwave-assisted extraction (MW) and Ultrasound-assisted extraction (US) in recent years. These new techniques in comparison to conventional methods increase the speed and efficiency of the process. There is an increasing demand for efficient extraction techniques that include shorter extraction time, less consumption of organic solvent, energy and cost saving. Advanced microwave extraction has developed a number of innovative techniques like vacuum microwave hydro-distillation, microwave-assisted solvent extraction, Soxhlet extraction, compressed air microwave distillation, solvent free microwave extraction. Microwave-assisted extraction has disadvantages which are related to non-homogenous heating but still, it has been accepted as a powerful and alternative method for the extraction of organic compounds. Until now, solvent extraction has been used for extraction of phenolics components from roses using methanol, ethanol or acetone as a solvent.

- **Extraction:** There are three main methods for the extraction of rose oil from the petals of plant material.
 1. Steam distillation
 2. Solvent extraction
 3. Superficial carbon-dioxide extraction
- 1. Steam distillation

In this method, an essential oil which is also known as rose otto or attar is produced. During this process, large stills usually of copper are filled with water and roses. This still is heated at 60-105 minutes. The rose oil obtained in the condensing apparatus is then collected in the flask. This distillation yields direct oil and concentrated oil. The water which condenses along with the oil is then redistilled to obtain water-soluble fractions of rose oil.

Rose Otto is clear light yellow in color and is mobile at room temperature. Formation of white crystals takes place which disappears when the oil is gently warmed. It has a very strong odor but when diluted gives a pleasant smell and is used as perfume. The hydrosol part of the distillate is called rose water and is a by-product which is used as flavors and in skin care.

2. Solvent extraction

In this method, flowers are extracted with the help of a solvent such as hexane that draws out aromatic compounds and other substances such as wax and pigments. For the removal of the solvent, vacuum processing is done so as to re-use the solvent. The waxy volume is called concrete which is then mixed with alcohol to dissolve aromatic constituents leaving behind the wax and other materials. The alcohol is evaporated at low pressure that leaves behind absolute which is further processed to remove the remaining impurities.

Rose absolute is deep reddish brown in color that forms no crystal.

3. Carbon-dioxide extraction

In this method, the Supercritical fluid CO₂ extracts the aromatic components from the plant material. It gives a wide range of compounds at low temperature providing the best results.

- **Separation:** Plant extract occurs as a mixture of chemical compounds with their different polarities. Therefore separation is a big challenge for their identification and characterization. The methods used for the separation of mixture include Column chromatography, TLC after which the separated products are then used for the determination of their structure.
- **Identification:** Identification of the separated compounds is done through spectroscopic techniques such as UV, IR, and NMR.

II. Materials and methods:

The petals of *Rosa rubiginosa* were collected in the month of March 2019 from the market. The separated petals were weighed around 500 gm. The method adopted for the extraction of chemical components from *Rosa rubiginosa* was solvent extraction. The Extraction was carried out in a round bottom flask using hexane as a solvent.

Part I: Isolation of Chemical components from the Petals of *Rosa rubiginosa*

Procedure:

The prewashed fresh petal (500 g) of *Rosa rubiginosa* was extracted using hexane as a solvent. Extraction was carried out for 24 hours by the solvent extraction method. The extract was refluxed at 40°C.



Fig. 1 *Rosa rubiginosa* petals



Fig. 2 Solvent extraction



Fig. 3 Rose absolute

Rose absolute: Rose absolute are similar to the essential oils. They are a highly aromatic and oil mixture. The chemical compound of rose absolute mainly includes citronellol, nerol, geraniol, eugenol, methyl eugenol, benzyl alcohol, β -phenethyl alcohol, nonadecane, and nonadecene.

Geraniol and citronellol occur frequently together. Due to their similar physical and chemical properties they cannot be separated by fractional distillation. According to the literature, the method for separation of geraniol and citronellol given by Tieman and Schmidt which depends on the phosphorous trichloride that acts on the alcohol in the ethereal solution. Geraniol is converted partly into hydrocarbon and partly into geranyl chloride. However, citronellol is converted to chlorinated acid ester of phosphorous acid which is soluble in alkalies and thus can be separated from the other compounds.

Benzyl alcohol and β -phenethyl alcohol are the simplest aromatic compound and are also chemically similar.

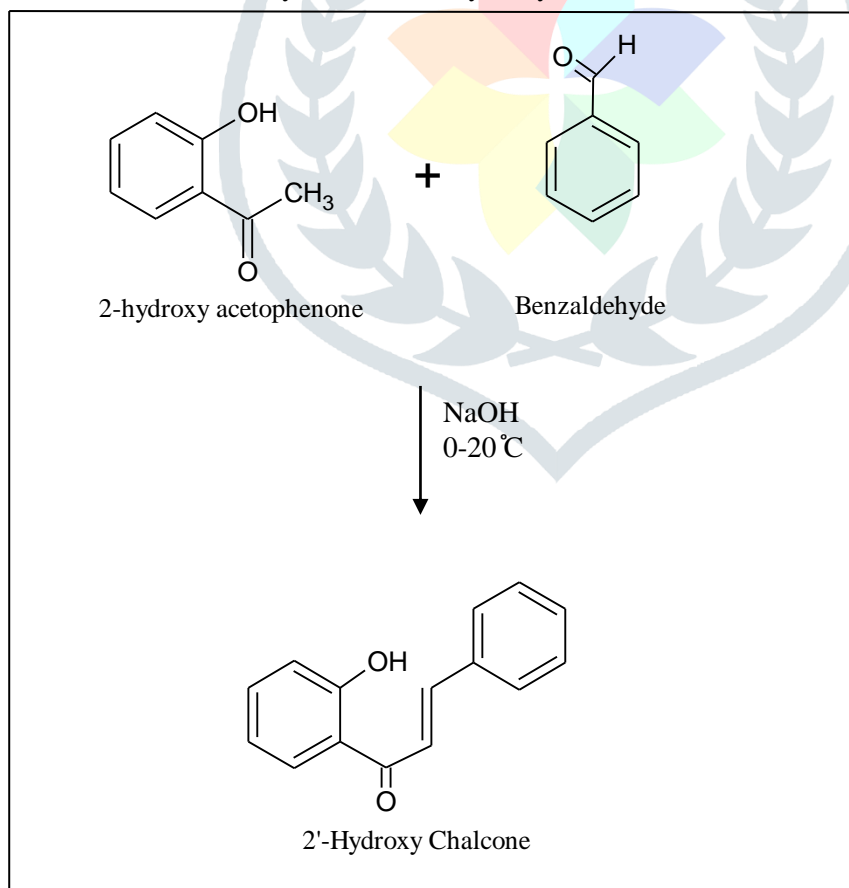
Efforts were made to separate different compounds by Column chromatography, TLC. However, in spite of our best efforts the mixture could not be separated.

Part II: Synthesis of 2'-Hydroxy Chalcone

Procedure:

Synthesis of 2'-Hydroxy Chalcone was carried out by using Aldol condensation. A solution of 20 ml 2-hydroxy acetophenone and 20 ml Benzaldehyde were mixed in the presence of base NaOH as a catalyst. The reaction mixture was kept at 0-20^o C temperature for 48 hours. The mixture was then filtered and washed with distilled water to remove access to raw materials. On completion of the reaction, it was monitored by TLC. Thin Layer Chromatography of reactants (Benzaldehyde and 2-hydroxy acetophenone) and the obtained product 2'-Hydroxy Chalcone was carried out in hexane: ethyl acetate (12:2). It was sprayed with 5% H₂SO₄ where brown color Spot of the final product was observed. The reactant mixture was acidified with HCl in an ice bath and the solid was then filtered and crystallized by ethanol. The yield of the obtained product 2'-Hydroxy Chalcone was 90%. The structure of the product was confirmed by comparing it with the authentic sample.

Table 1.1 Synthesis of 2'-Hydroxy Chalcone



III. Results and Discussion: (Part I)

IV. Conclusion:

1. Four components were obtained from the Petals of *Rosa rubiginosa*.
2. Rose absolute was obtained by solvent extraction of these petals using Hexane,.
3. Three components, Geranial, Citronellal, and rose oil were also identified. Geranial and Citronellal were identified as aldehyde by the Schiff reagent test.
4. 2'-Hydroxy Chalcone was synthesized by using Aldol condensation and its structure was established.

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