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RESEARCH PAPER

Synthesis novel flavone from vanillin

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Abstract: Flavone compounds present as secondary metabolites in many plants have beneficial phytochemical activity. Isolation of flavones from various parts of plants has been widely carried out, but the synthesis pathway is another way to obtain higher yields. This research aimed to synthesize flavone compounds from vanillin and 2-hydroxyacetophenone through chalcone intermediates to produce a 74% yield using an iodide catalyst. This new flavone compound has been used as a chemosensor to detect anions.

Keywords: synthesis, Flavone, vanillin

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INTRODUCTION

Flavon (*flavus*) is a class of Flavonoids based on the backbone structure of 2-phenylchrome-4 [1]. It is a flavonoid group often found in leaves, fruits, and flowers in the form of glucosides. It plays a role in giving color and taste to seeds, flowers, and fruit and aroma to plants. It also protects plants from environmental influences as antimicrobials and protects against exposure to UV rays [2]. The main source of flavone group compounds comes from plants. The phytochemical activity has resulted in many isolations being carried out from various related plant sources.

The 3,3',4',5,7-pentahydroxy flavone Compound (Quercetin) is a compound derived from the Flavonoid group with activity as an anti-pain and anti-inflammatory [3]. Flavone derivative compound 5,7,4'-trihydroxy-3'(3-methylbut-2-enyl)-3-methoxyFlavone has been isolated from the leaves of M. involucrata (Roxb.) Baill [4]. Flavones have also been isolated from corncob ethyl acetate extract [5] and *green peel seed* ethanol extract [6]. Flavones from the leaves of C. album are used as poultices for insect bites, sunburns, and arthritic joints and as a mild laxative [7]. Flavonoids and Glycosides Flavonoids from Myrsine africana and their inhibitory activity against fungal tyrosinase have been isolated by Kishore et al. [8], and flavones from water hyacinth have been isolated by Elvira et al. [9]. Ratna and Chatarina [10] isolated flavones from dewa leaves, flavones from Euphorbia neriifolia leaves [11], Flavones from *mahogany* seeds [12], Flavones from Zingiberaceae rhizome root extract [13].

Isolation of flavone compounds directly from plant sources often produces low yields but requires large amounts of raw materials. Therefore, using available chemical materials, an alternative to obtaining Flavone compounds with greater yields is taken from the synthesis method. Such as the synthesis of Flavones from 2-hydroxyarylchalcones by Patel and Shah [14], Flavones from 2'-alyloxy-α, β-dibromochalcone and iodides [15], Flavones from o-benzyloxyacetophenone as therapy (anti-diabetic, anti-inflammatory, antioxidant, and anti-cancer) [16].

Flavone compounds, because they have an aromatic structure and the phi bond electrons are located alternately (conjugated), undergo PET (photoinduced electron transfer) so that when they meet anions, they induce the aromatic side so that a color change or fluorescence occurs in the solution. Therefore flavone compounds can be used as a chemical detection tool (chemical sensor) to detect the presence of anions in solution. So to get flavone compounds that can act as chemosensors with high sensitivity, reactants with conjugated aromatic characteristics, such as vanillin compounds, must be used.

This research has synthesized Flavone compounds from vanillin as the basic ingredient (via an aldol condensation reaction with 2-hydroxyacetophenone to produce chalcone) with an iodide catalyst. This flavone compound has been tested to detect F^- ions [17][18]. Sensors based on vanillin have also been used to detect another anion, cyanide anion [19][20].

MATERIALS AND METHODS

Materials

Ingredients: chalcone (E)-3-(hydroxy-3-methoxyphenyl)-1-(2-hydroxyphenyl) prop-2-en-1-on) synthesized, o-hydroxyacetophenone Meck p.a grade, Meck iodide p.a grade, DMSO Merck p.a grade, ethanol Merck p.a grade, distilled water.

Tools

Reflux tool set, hotplate, magnetic stirrer, Buchner filter, analytical balance (Libror EB-330 Shimadzu), melting point determination tool (Electrothermal-9100), infrared spectrophotometer (Shimadzu Prestige-21), gas chromatography-mass spectrophotometer (Shimadzu QP-2010S), 1H-NMR spectrophotometer (JEOL JNM ECA-500, 500MHz), and 13C-NMR spectrophotometer (JEOL JNM ECA-500, 125MHz).

Method.

10 mmol of chalcone, 15 mL of DMSO, and 1 gram of I2 were put into the reaction flask. Then the mixture was refracted at 180°C for 1.5 hours [21] and poured into 100 mL of cold distilled water. The precipitate formed was then recrystallized with ethanol. The solid obtained was determined for its melting point and characterized by FT-IR, mass spectra, 1H-NMR, and 13C-NMR.

RESULTS AND DISCUSSION

The synthesis scheme is presented in Figure 1.

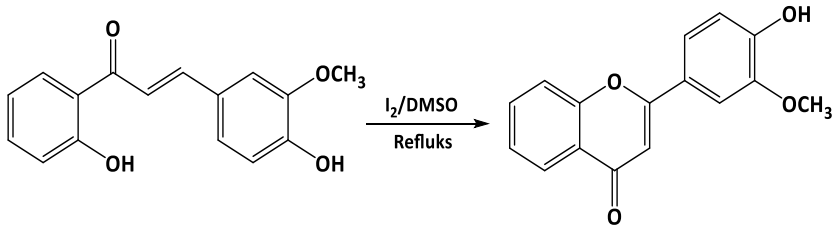

Figure 1. Flavone Synthesis Pathways

The synthesized product is a yellowish cream-colored powder with a yield of 74%, a molecular weight of 268 g/mol, and a melting point of 189-192 °C. The synthesized product's structure was elucidated using the FT-IR spectrometer instrument, whose spectra are presented in Figure 2

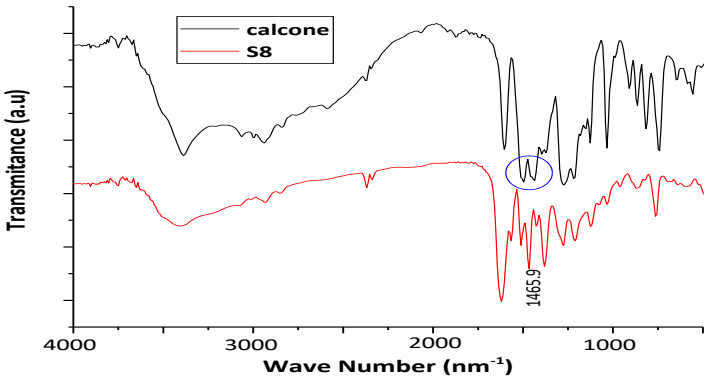

Figure 2. FT-IR spectra of flavone synthesis (code S8) from chalcone

The FT-IR spectra show that the reaction for the formation of Flavone compounds has gone well. This is evidenced by the appearance of C=O (ring R3) absorption at 1620 cm¯1, C=C aromatic (rings R1, R2, and R3) at 1465 cm¯1. Confirmation of the structure of the synthesized product was also carried out with a 1H-NMR spectrometer whose spectra are presented in Figure 4.

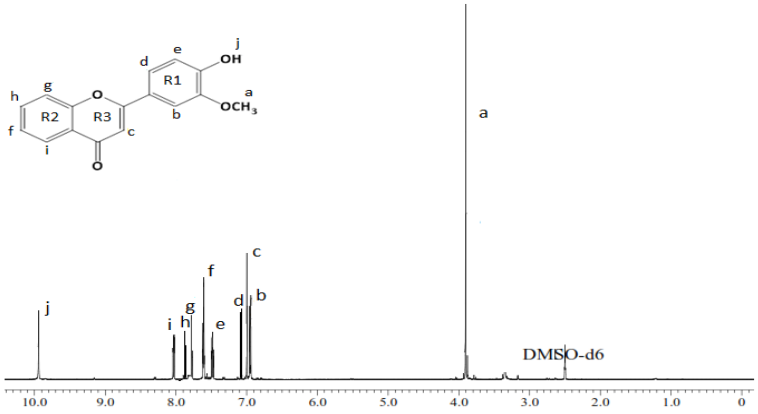

Figure 3. 1H-NMR Spectra Flavone synthesized from chalcone

The O-H proton and 2 alkene protons from the chalcone, which appear at (based on the chem draw theory, Figure 5) δ 16.47 ppm, 7.0 ppm, and 8.06 ppm respectively, disappear from the spectra, replaced by the emergence of protons from the ring Flavone (Hc) at δ 6.69 ppm and fused cyclic benzene ring proton (Hh) experienced a downfield from 6.96 ppm to 8.04 ppm. The O-H proton of the chalcone phenol ring, which appeared at δ 3.83 ppm (confirmed with the H-NMR spectra of chalcone in Figure 4), experienced a downfield to δ 9.94 ppm in Flavone (the loss of OH and the formation of a C=O bond in the R3 ring).

Further confirmation was also carried out with a mass spectrometer whose spectra were presented in Figure 5.

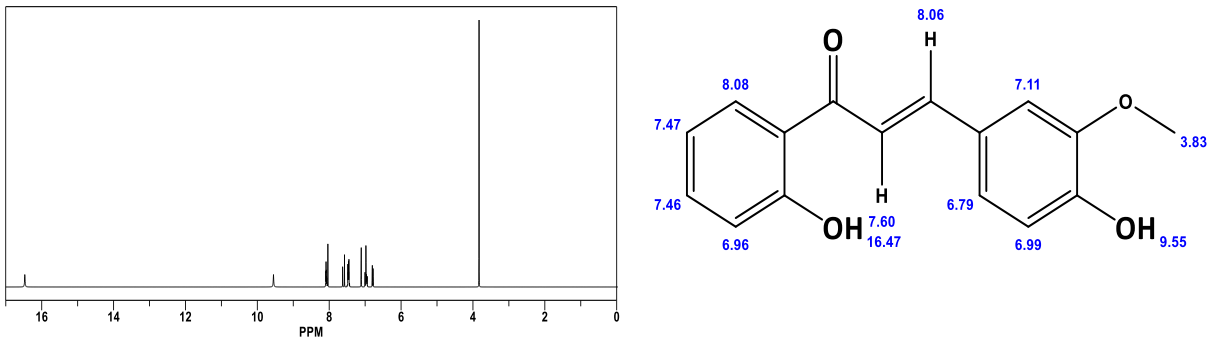

Figure 4. Theoretically chalcone H-NMR spectra

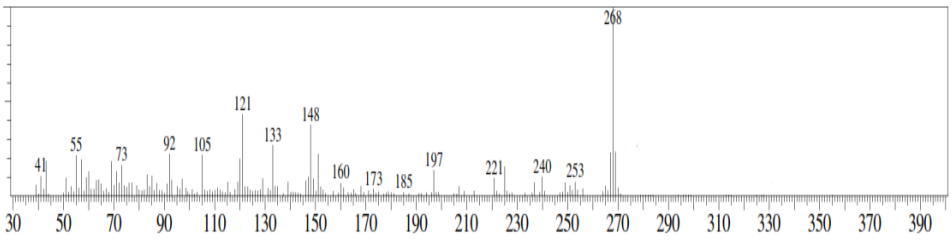

Figure 5. Mass spectra of Flavones synthesized from chalcone

The peaks that appear in the mass spectra indicate the peaks of the fragment compounds from the flavone compounds formed, and the numbers at the peaks indicate the molecular mass of the fragments. The pattern of fragmentation of flavone compounds is presented in Figure 6.

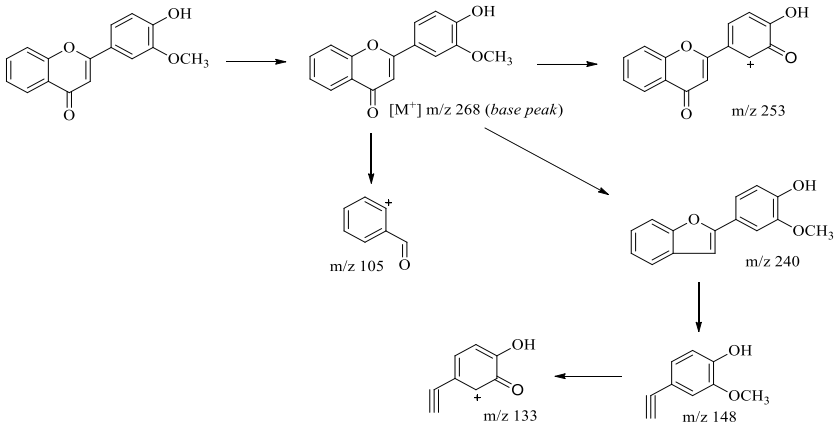

Figure 6. Flavone fragmentation patterns

Further confirmation of the elucidation of the structure of the synthesized product was carried out with a 13C-NMR spectrometer whose spectra were as shown in Figure 7. The alkene carbon in the chalcone, which experienced a shift at δ 146.1 ppm and 118.7 ppm (Figure 8), disappeared from the spectra, and new spectra appeared at δ 105.18 ppm (Cb) and 163.00 ppm (Co) due to the formation of the Flavone ring.

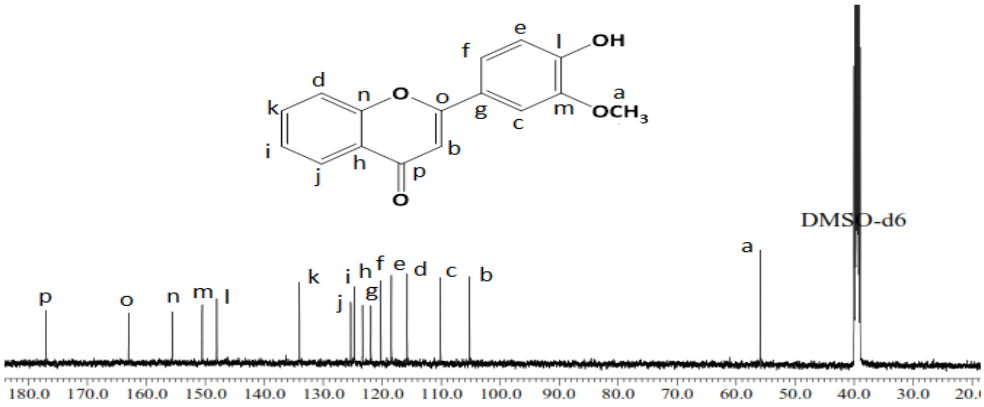

Figure 7. 13C-NMR spectra of flavone compounds

Figure 8 presents the peaks of the flavone spectra theoretically as a comparison that the formation of new C-C bonds belonging to Flavone.

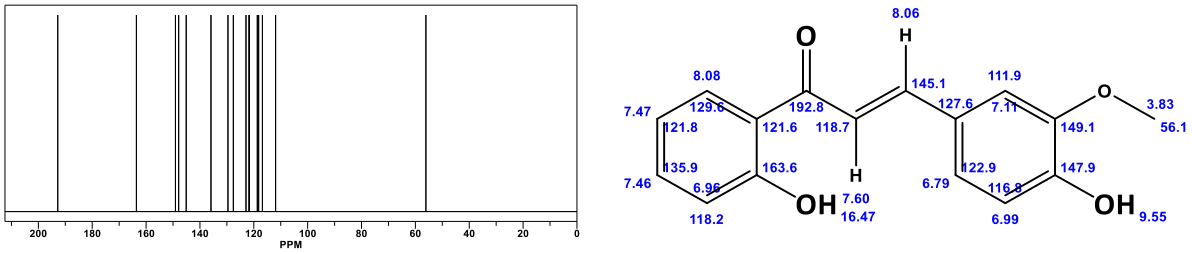

Figure 8. Theoretical spectra of flavone

Comparing the results of the HNMR and CNMR spectra theoretically, the instrument measurements of the flavone compounds formed indicate a match in the amount and absorption value. It shows that the result of the synthesis can be ascertained as the flavone compound that is sought.

CONCLUSION

Despite the widespread use of flavone isolation from diverse plant sections, larger

quantities can also be achieved using the synthesis process. Synthesis of Flavone compounds from vanillin with an iodide catalyst gives a good yield of 74%.

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