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ISOLATION, IDENTIFICATION AND SYNTHESIS OF ANALOGUES OF KAEMPFEROL ISOLATED FROM PHYLLANTHUS NIRURI

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Abstract: Kaempferol has been isolated from *Phyllanthus niruri* and identified. Five analogues of Kaempferol have been synthesized and characterized by chemical and spectral data.

Keywords: Kaempferol, Analogues.



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INTRODUCTION

Flavonoids have been reported to show variety of biological activities including antimicrobial [1], cytotoxicity [2], anti-inflammatory [3] as well as antitumor activities [4]. The work of Hertog and co-workers showed the inverse correlation between flavonoids intake and coronary heart disease mortality [5]. Flavonoids have attracted the interest of researchers because they are powerful antioxidants which can protect the human body from free radicals [6-8]. Kaempferol is the Flavonoid which is the bioactive ingredient of medicinal plants. Therefore it was thought worth experimenting to isolate and characterize this bioactive antioxidant from the plants of genus *Phyllanthus* and to synthesize the analogues of the isolated Kaempferol.

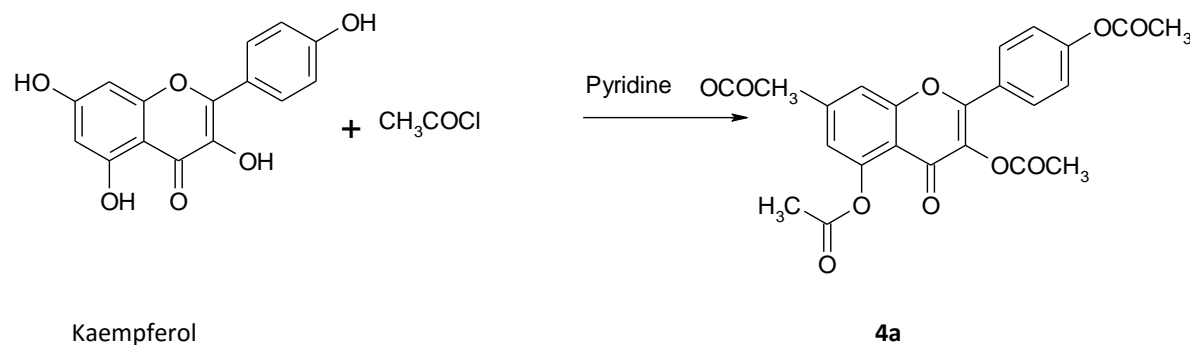
MATERIAL AND METHOD

Kaempferol was isolated from the plant of *Phyllanthus niruri* and identified by TLC, UV and NMR analysis and following analogues have been synthesized

Kaempferol Acetate (4a)

3,4',5,7-Tetra-O- Acetyl Kaempferol

Kaempferol (0.001 mole, 0.28 g) was dissolved in a mixture of dimethyl formamide (20 ml) and pyridine (2 ml). This solution was kept in ice bath at 5°C. Acetyl chloride (0.004 mole, 0.312g) was slowly added with constant stirring. The mixture was allowed to stand at room temperature for 24 hours. The mixture was poured on crushed ice. The solid so obtained was filtered off, washed with cold water and dried. The crude product was crystallized from methanol as white needles. Yield 74%, Melting point 178°C



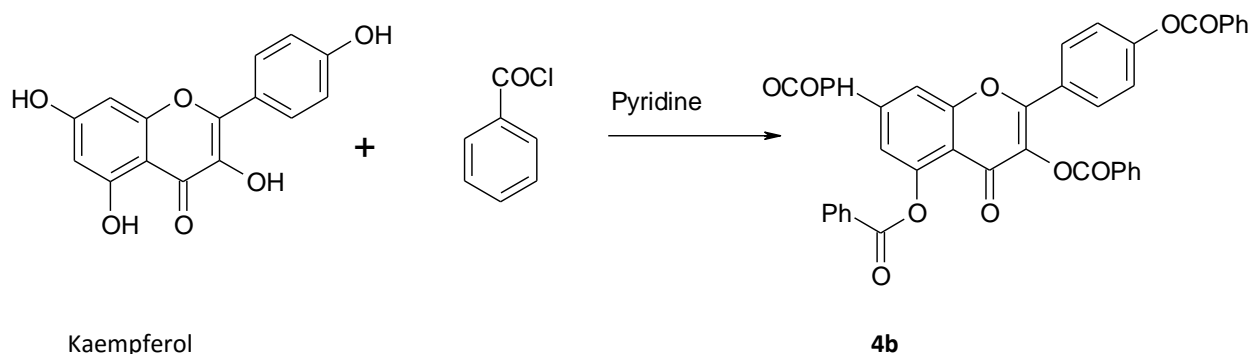
3,4',5,7-Tetra-O- Acetyl Kaempferol

NMR δ 1.12(6H, s, OCOCH₃), δ 3.4 (6H, s, OCOCH₃), δ 6.18 (1H, d, ArH), δ 6.39 (1H, d, ArH), δ 6.88 (2H, d, ArH), δ 7.55 (2H, d, ArH),

Kaempferol Benzoate (4b)

3,4',5,7-Tetra-O- Benzoyl Kaempferol

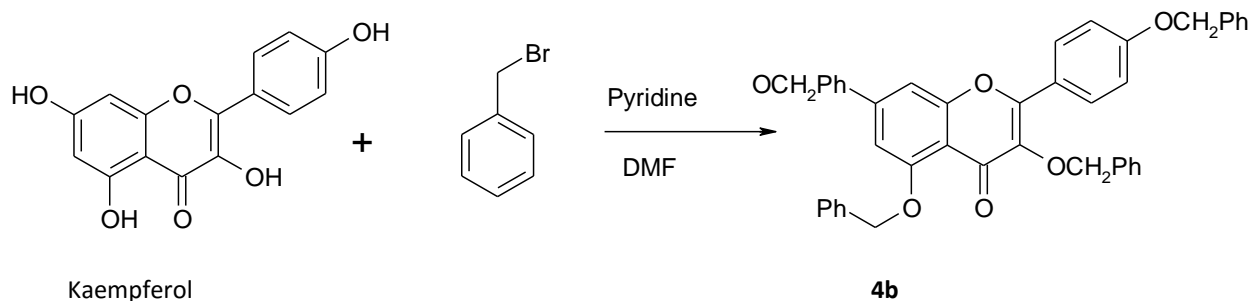
Kaempferol (0.001 mole, 0.28 g) was dissolved in a mixture of dimethyl formamide (20 ml) and pyridine (2 ml). This solution was kept in ice bath at 5°C. benzoyl chloride (0.004mole, 0.56g) was slowly added with constant stirring. The mixture was allowed to stand at room temperature for 24 hours. The mixture was poured on crushed ice. The solid so obtained was filtered off, washed with cold water and dried. The crude product was crystallized from glacial acetic acid as yellowish crystals. Yield 61 %, Melting point 164 °C



3,4',5,7-Tetra-O- Benzoyl Kaempferol

3,4',5,7-Tetra-O- Benzyl Kaempferol (4c)

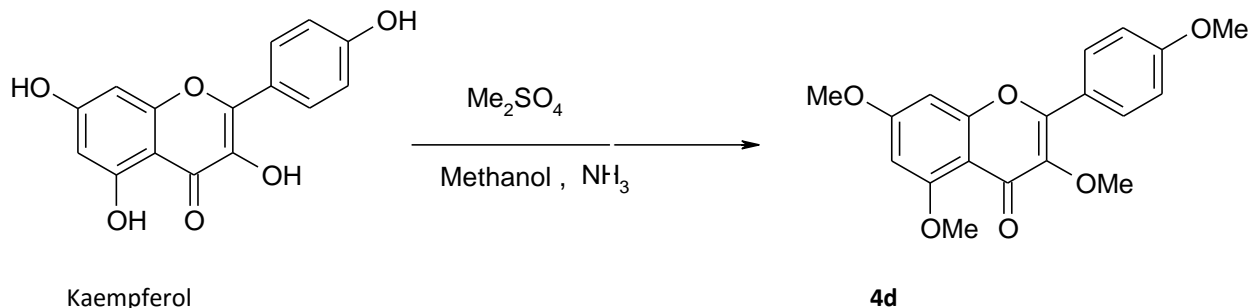
Kaempferol (0.001 mole, 0.28 g) was dissolved in a mixture of dimethyl formamide (20 ml) and pyridine (2 ml). This solution was kept in ice bath at 5°C. benzyl bromide (0.004mole, 0.684g) was slowly added with constant stirring. The mixture was then refluxed for 2 hours. The mixture was then allowed to cool and poured on crushed ice. The solid so obtained was filtered off, washed with cold water and dried. The crude product was crystallized from glacial acetic acid as yellowish crystals. Yield 65 %, Melting point 178 °C



3,4',5,7-Tetra-O- Benzyl Kaempferol

3,4',5,7-Tetra-O- methyl Kaempferol (4d)

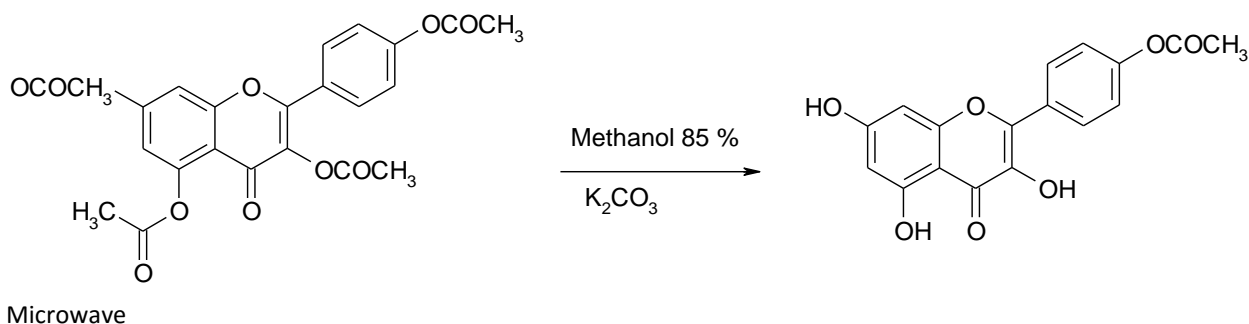
Kaempferol (0.001 mole, 0.28 g) was dissolved in methanol (20 ml) and liquid ammonia (2 ml). To this mixture dimethyl sulphate (0.004 mole, 0.504g) was added slowly and the mixture was refluxed for four hours. The mixture was allowed to cool and poured on crushed ice. The solid so obtained was filtered off, washed with cold water and dried. The crude product was crystallized from methanol as colorless crystals. Yield 65 %, Melting point 162 °C.



3,4',5,7-Tetra-O- methyl Kaempferol

Acetyl Kaempferol (4e)

A mixture of kaempferol teracetate 4a (0.001 mole, 0.454 g), anhydrous potassium carbonate in catalytic amount and methanol 85% (50 ml) was kept in microwave for 3 min. The reaction mixture was allowed to cool and concentrated. The solid so obtained on cooling was filtered off and crystallized from ethanol as white crystals. Yield 60 % and melting point 158°C.



Kaempferol Acetate

4e

Acetyl Kaempferol

¹H NMR (Plate No 4e) : δ 2.04(3H, s, OCOCH₃), δ 6.89 (1H, d, ArH), δ 7.23 (1H, d, ArH), δ 7.23 (2H, d, ArH), δ 9.25 (1H, s, OH), δ 9.87 (1H, s, OH), δ 12.46 (1H, s, OH),

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