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# First isolation of a dihydroflavonol from the Sudanese material of *Pulicaria crispa* forsk

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**Abstract**
An investigation of the chemical constituents of *Pulicaria crispa* (Compositeae) led to the isolation a dihydro flavonol. The isolation of the dihydroflavonol was carried out using chromatographic techniques, and the chemical structure was established by spectral data (IR, UV, $^1$HNMR and MS).

**Keywords:** *Pulicaria crispa (Forsk.),* Flavonoids, Isolation, Characterization

## 1. Introduction
Flavonoids are widely distributed phenolic compounds. They occur in all parts of plants as complex mixtures of different components. Their structure consists of two aromatic rings joined by a three carbon link (a $C_6$-$C_3$-$C_6$ configuration). The aromatic rings are usually oxygenated rendering the flavonoids hydrogen and electron donors. Flavonoids are believed to be endowed with biological activities, such as anti-inflammatory, anti-allergic, anti-platelet, immunomodulatory and anti-tumoral activities (Craig, 1999; Ielpo *et al*., 2000; Prior, 2000) [7, 11, 12].
*Pulicaria crispa* is distributed in Saudi Arabia, Kuwait, Iran, Iraq, Egypt, Afghanistan, Pakistan, India and parts of north and west tropical Africa (Boulos, 2002; Al-Rawi, 1987) [4, 3]. *Pulicaria crispa* is a medicinal plant used by people of southern Egypt and Saudi Arabia to treat inflammation and also as an insect repellent (Ross *et al.*, 1997) [13]. It is also used as a herbal tea. Phytochemical studies of this herb revealed that the plant is a rich source of sesquiterpene lactones of the guaianolide (Dendougui *et al*., 2000) [9], eudesmanolide and xanthanolide classes as well as diterpenes. Amino acids, carbohydrates, coumarins, flavonoids, glycosides, proteins, sterols, tannins, and volatile oil were also detected (Abd-Alla, 2004) [1].
The immunostimulatory effects of the methanolic extract of *Pulicaria crispa* were investigated in mice before and after infection with *Schistosoma manson* with promising results (Awad *et al*., 2001) [2].

## 2. Materials and methods
### 2.1 Materials
IR spectrum was recorded on a JASCO FTIR- 6100 spectrophotometer. The UV absorption was recorded on a Shimadzu UV spectrophotometer model – 240. $^1$HNMR spectra were measured on a Jeol EX-500 spectrophotometer operating at a frequency of 500 MHz using DMSO-*d*6 as solvent.

### 2.2 Plant materials
The aerial parts of *Pulicaria Crispa* were collected in August 2011 from White Nile (Sudan) during the flowering stage, and was kindly identified by Dr. Hiddar A. Elgadir at the Herbarium of the medicinal and aromatic plants research institute (MAPRI), Khartoum, Sudan.

## 3. Methods
### 3.1 Extraction and isolation of flavonoids
Dried and finely powdered aerial parts of *Pulicaria Crispa* (2Kg) were extracted with 70 % methanol at room temperature. The solvent was evaporated *in vacuo* to yield a dark amorphous mass (170 g). The methanolic extract was purified by a polyamide 6S column using $H_2O$/ MeOH mixtures of decreasing polarities as eluting solvent to give 50 fractions. The fractions were visualized under the UV light at both long and short wave lengths at 254 and 365nm using PC solvent systems, similar fractions were pooled together. Therefore, we focused on the isolation of flavonoid constituents.
Fractions (20-24) were subjected to further purification on a sephadex LH-20 column eluted with saturated butanol. The required fractions were evaporated *in vacuo* and the isolated compound – compound A, was recrystallized from methanol to give colourless crystals (30mg). Purity was checked by TDPC technique.

## 4. Results and discussion
The IR spectrum of compound A showed ν (cm-1, KBr disc) 3429 (OH), 2920 (aliphatic C -H), 1642 (C=O), 1574 -1455 (C= C, Ar.), 1000 (C- O), below 900 cm$^{-1}$ (C-H, Ar, bending). This compound cannot be an anthocyanin or a catechin since the IR spectrum gave a carbonyl stretching at 1642 cm$^{-1}$. It could be: aflavonol, a flavanone, a flavone, a chalcone, an aurone, an isoflavone, a dihydroflavonol or a dihydrochalcone.
The UV spectrum showd $\lambda_{max}$ (methanol): 289 nm. Such absorption is characteristic of isoflavones, flavanones and dihydroflavonols which are characterized by absence of conjugation between the A and B rings.
Addition of the UV shift reagent sodium methoxide to a methanolic solution of this compound gave a bathochromic shift with decrease in intensity suggesting the presence of a 3

– OH and indicating that this compound could be a dihydroflavonol. The sodium acetate spectrum did not reveal any bathochromic shift indicating absence of free 7-OH function. The aluminum chloride spectrum gave a 10nm bathochromic shift. No change in the spectrum was observed when HCl was added. This cites evidence for the absence of catechol moieties and the presence of a 5-OH function.

The $^1$H NMR (500MHz, DMSO-$d_6$) spectrum (Table 1) showed δ3.74 (3H) due to a methoxyl group. The resonances at δ6.07(1H), 6.72(1H) are due to $C_6$- and $C_8$- protons respectively, while the signal at δ6.84 accounts for the B ring protons which usually resonate at lower field in contrast to A ring protons. (Harborne, 1994).

**Table 1:** $\delta_H$ of compound Vd

| δ(ppm) | proton |
|---|---|
| 3.74(s) | methoxyl |
| 4.98(s) | Anomeric proton |
| 6.07(d) | $C_6$ –H |
| 6.72(d) | $C_8$-H |
| 6.84(s) | B-ring protons |

The mass spectrum gave the ion m/z318 for $M^+$+ 2H. Other important fragments (m/z166 and m/z151) resulting from the retro Diels-Alder fission (scheme I) site evidence for the following tentative structure:

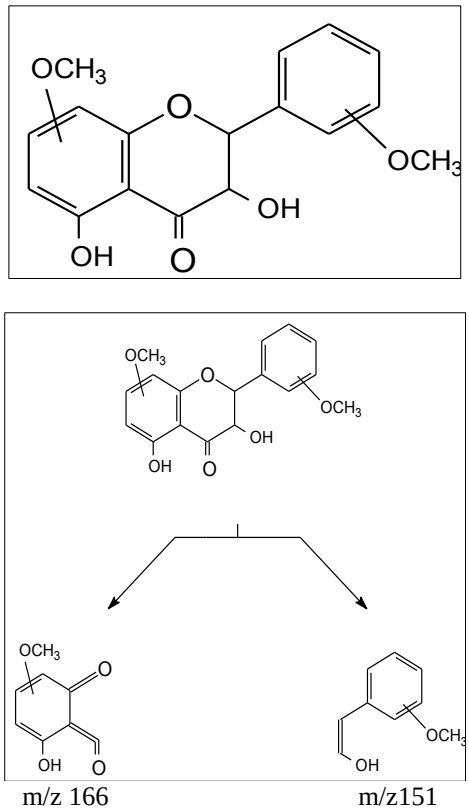


**Fig 1:** Retro Diels-Alder fission of compound A

**5. Acknowledgements**

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