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Trans-isoferulic acid from *Curcuma longa*

[Ácido *trans*-isoferulico en *Curcuma Longa*]

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Abstract

Trans-isoferulic acid was isolated from the carbon tetrachloride soluble fraction of a methanol extract of the rhizomes of *Curcuma longa* (Zingiberaceae). The structure of the isolated compound was elucidated by comprehensive analysis of spectroscopic data. This is the first report of its occurrence from this plant.

Keywords: *Curcuma longa*; Zingiberaceae; *trans*-isoferulic acid

Resumen

El ácido *trans*-isoferulico fue aislado de la fracción soluble en tetracloruro de carbono del extracto metanólico de los rizomas de *Curcuma Longa* (Zingeraceae). La estructura del compuesto aislado fue elucidada por análisis de los datos espectroscópicos. Este es el primer reporte de su presencia en esta planta.

Palabras Clave: *Curcuma longa*; Zingiberaceae; ácido *trans*-isoferulico

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INTRODUCTION

Curcuma longa (Family- Zingiberaceae, Bengali name- Halud) is a perennial herb that measures up to 1 m high with a short stem and distributed throughout tropical and subtropical regions of the world. It is widely cultivated in Asiatic countries such as Bangladesh, India and China (Araujo and Leon 2001). The multicomponent essential oils of turmeric have anti HIV (De Clercq 2000), antibacterial (De *et al*., 2009) and antioxidant (Singh *et al*., 2010) properties. Curcumin, a hydrophobic polyphenol derived from the rhizomes of *C. longa* possesses antioxidative, anticarcinogenic (Bar-Sela *et al*., 2010), anti-proliferative, anti-inflammatory (Ravindran *et al*., 2010) and hypolipidemic activities (Babu and Srinivasan 1997). Previous phytochemical studies with *Curcuma* species led to the isolation of several sesquiterpenes such as wenyujinlactone A, neolita-mone A, zedoarondiol, isozedoarondiol, aerugidiol, curcumol, curdione, (1R,10R)-epoxy-(-)-1,10-dihydrocurdine (Wang *et al*., 2007) and parviflorene F (Ohtsuki *et al*., 2008) and some curcuminoids e.g., curcumin, demethoxycurcumin and bisdemethoxycurcumin (Pozharitskaya *et al*., 2008).

We herein report the isolation of *trans*-isoferulic acid for the first time, from the carbon tetrachloride soluble fraction of a methanol extract of *C. longa*.

MATERIALS AND METHODS

General experimental procedure

The ^{1}H NMR spectrum was recorded using a Bruker AMX-400 (400 MHz) instrument and the spectrum was referenced to the residual nondeuterated solvent signal. Preparative Thin Layer Chromatography (PTLC) was carried out using Merck Si gel 60 F_{254} on glass plates (20 cm X 20 cm) at a thickness of 0.5 mm. TLC was conducted on normal-phase Merck Si gel 60 F_{254} on glass plates and the spots on TLC and PTLC plates were visualized under UV light at 254 nm as well as by spraying with vanillin sulfuric acid followed by heating for 5 minutes at 110 ºC.

Plant Material

Rhizomes of *C. longa* were collected from Dhaka in February 2008. A voucher specimen for this collection has been deposited in the herbarium of the Department of Botany, University of Dhaka. The samples were cut into small pieces and sun dried for 7 days followed by oven drying for 24 hours at 40 °C to facilitate grinding.

Extraction and isolation

The powdered material (533 g) was soaked in 1.5 L of methanol in a large conical flask for 7 days with occasional shaking and stirring. The whole mixture was then filtered off through a cotton plug followed by Whatman filter paper no.1 and the filtrate thus obtained was concentrated at 40°C with a rotary evaporator. A portion (5.0 g) of the concentrated methanol extract was fractionated by the modified Kupchan partitioning protocol (Vanwagenen *et al*., 1993) to yield petroleum ether (1.0 g), carbon tetrachloride (1.1 g), dichloromethane (0.85 g) and aqueous (1.65 g) soluble materials.

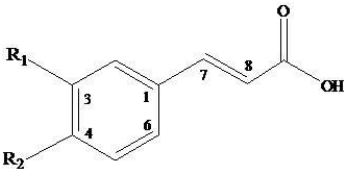
An aliquot of the carbon tetrachloride soluble partitionate (650 mg) was fractionated by column chromatography (CC) over silica gel (Kieselgel 60, mesh 70-230) using petroleum ether and ethyl acetate mixture in order of increasing polarities. A total of 143 fractions were collected, each 20 ml. PTLC of column fractions 91 to 96 eluted with 50% ethyl acetate in petroleum ether over silica gel using 2% methanol in dichloromethane as the developing solvent provided compound **1** (yield - 4.5 mg).

RESULTS

Repeated chromatographic separation and purification of the carbon tetrachloride soluble partitionate of a methanol extract of the rhizomes of *C. longa* provided *trans*-isoferulic acid, the structure of which was solved by NMR analysis and by comparison with published values.

Compound 1: *trans*-isoferulic acid (4.5 mg, 0.09% yield): yellow powder; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (1H, *d*, *J* = 16.0 Hz, H-7), 7.03 (1H, br. *d*, *J* = 8.0 Hz, H-6), 6.99 (1H, br. *s*, H-2), 6.83 (1H, *d*, *J* = 8.0 Hz, H-5), 6.41 (1H, *d*, *J* = 16.0 Hz, H-8), 5.72 (1H, br. *s*, H-3), 3.86 (3H, br. *s*, OCH_3-4).

Figure 1. structures of *trans*-isoferulic acid (**1**) and *trans*- ferulic acid (**2**)



1: R_1 = -OH R_2 = -OMe
2: R_1 = -OMe R_2 = -OH

DISCUSSION

The ^{1}H NMR spectrum of compound **1** displayed a singlet of three proton intensity at δ 3.86 demonstrative of the presence of a methoxyl group at C-4. It also showed a broad singlet at δ 6.99 (H-2) and a doublet (J = 8.0 Hz) centered at δ 6.83 (H-5) and a broad doublet (J = 8.0 Hz) at δ 7.03 (H-6) typical for a 1,3,4-trisubstituted aromatic moiety in compound **1**. The doublets (J = 16.0 Hz) centered at δ 7.50 and 6.41 could be assigned to the *trans* coupled protons H-7 and H-8, respectively. The relatively low field resonance of H-7 could easily be explained by its beta (β) position to the carbonyl group, in the form of a carboxylic acid.

Co-TLC of compound **1** with *trans*-ferulic acid (**2**) previously isolated from the same extract showed two distinct spots having different R_f values. This indicated that compound **1** was a structural isomer of *trans*-ferulic acid. Thus, it was characterized as *trans*-isoferulic acid (Figure 1). The identity of compound **1** was further substantiated by comparison of its spectral data with literature values (Prachayasittikul *et al*., 2009). This is the first report of isolation of *trans*-isoferulic acid from *C. longa*.

CONCLUSION

The present phytochemical study of the carbon tetrachloride soluble fraction of the methanol extract of *C. longa* afforded a phenylpropanoid derivative, the structure of which was established as *trans*-isoferulic acid extensive spectroscopic studies as well as by comparison with published results.

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