

COMPOSITION OF ESSENTIAL OIL ISOLATED FROM MARIGOLD (*TAGETES ERECTA* L.) FLOWERS CULTIVATED IN LAHORE, PAKISTAN

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Keywords: Essential oil, GC-MS, Marigold, Pakistan

Abstract

The composition of essential oil of marigold (*Tagetes erecta* L.) flowers was investigated. The oil was analyzed by GC-MS for identification of various components. Among these, 3-cyclohexene-1-methanol, α , α ,4-trimethyl-, acetate was the most abundant compound (31.67%) followed by phenol, 2,4-bis (1,1-dimethylethyl)- (14.35%) and eucalyptol (14.27%). Other compounds included dodecanoic acid (5.97%), tetradecanoic acid (5.58%), octadecane (4.93%), propanoic acid, 2-bromo-2-methyl-, ethyl ester (5.43%), cyclohexanol, 1-methyl-4-(1-methylethanyl)-, acetate (5.33), *n*-hexadecanoic acid (4.70), *p*-menth-1-en-8-ol (4.20%) and thymol (3.57%).

Introduction

Tagetes erecta L. belonging to Asteraceae, is native to Mexico and has medicinal, pharmaceutical, aromatic and ornamental properties (Abbas *et al.* 2019). It is an annual drought resistant plant grown widely because of its beautiful flowers and easy availability throughout the year. It is a commercially exploited floriculture crop of Pakistan planted in bedding of landscape areas (Zulfiqar *et al.* 2020). Plant possesses large size cut flowers, which are different in shape, color and size. The flowers are sold in the market as loose flowers and highly suitable for beautification (Aslam *et al.* 2016). It is used as a flavoring agent and an edible dye as a substitute of saffron, which yields the yellow color (Sowndharya and Giri 2020). It has antioxidant, nematocidal and phenolic compounds, which are used in the preparation of medicines (Ayub *et al.* 2017).

Essential oils (EOs) play a vital role in plants by producing a specific aroma, flavor and to protect the plant from pathogens (Perczak *et al.* 2019). These characteristics, together with their diverse biological activities have attracted high interest from medicine, perfumery and food processing industry (Ayaz *et al.* 2017). *T. erecta* flowers are a rich source of secondary metabolites such as carotenoids, lutein, triterpenes, thiophenes and flavonoids (Kazibwe *et al.* 2017). Essential oil prepared from *T. erecta* flowers possesses complex composition of aromatic and volatile components which exhibited a wide range of anti-inflammatory, antinociceptive, antioxidant, insecticidal, antifungal, anticancer, antiepileptic, allelopathic, larvicidal, hepatoprotective, anti-diabetic, antidepressant, wound healing and mosquitocidal activities (Safar *et al.* 2020). Therefore, the present study was undertaken to assess the composition of essential oil of marigold flowers cultivated in Pakistan.

Materials and Methods

The present research work was carried out at the Faculty of Agricultural Sciences, University of the Punjab, Lahore, Pakistan; and Pakistan Council of Scientific and Industrial Research (PCSIR) Laboratories, Lahore, Pakistan. Full bloomed, healthy looking fresh flowers of marigold

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were plucked from Punjab University Lahore. Plucking of flowers was done early in the morning to avoid any possible loss of essential oil. Paper boxes were used to transfer flowers into the laboratory for further study process. Only the flower petals were used for the recovery of essential oil. Methanol was used as organic solvent in the Soxhlet's extraction apparatus. Volatile oil was sucked up by siphon in the thimble along with methanol through the condensers. The process was repeated 3-4 times. After taking out whole aroma out from the flowers, rotary evaporator was used for the distillation of recovered solvent.

The oil was analyzed for its chemical components. Gas chromatography 6890 Agilent /mass spectroscopy 5973 Agilent were used for identification of various constituents of essential oil. To obtain the chemical composition, sample of the extracted oil was injected with a syringe into the GC and was run as per set conditions: oven initial temperature was 50 °C, column DB-5 (30 m × 0.3 μm × 0.25 μm), ramping: 7 °C/min till 230 °C, final temperature was 230 °C for 5 min. Injector temperature was 180 °C, carrier gas was helium, flow rate 1.0 μl/min, split less, volume 0.3 μl, μS: source temperature 230 °C, Quad 150, 70 ev, EI. MS was done by comparing the chromatogram with library.

Results and Discussion

GC-MS chromatogram indicated that there were 11 constituents in essential oil of marigold (Fig. 1). Details of these compounds regarding their retention times, peak area percentages, molecular formulae and molecular weights are presented in Table 1. Among these, 3-cyclohexene-1-methanol, α , α ,4-trimethyl-, acetate (31.67%), also known as α -terpinyl acetate, was the principal constituent of the essential oil of marigold. In contrast to the present study, Gutterrez *et al.* (2006) reported piperitone (19.2%) followed by β -caryophyllene (15.2%) as the major components of essential oil in flowers of marigold collected from Mexico. Likewise, Krishna *et al.* (2004) also found piperitone (28.5%) followed by piperitenone (10.9%) as the major components

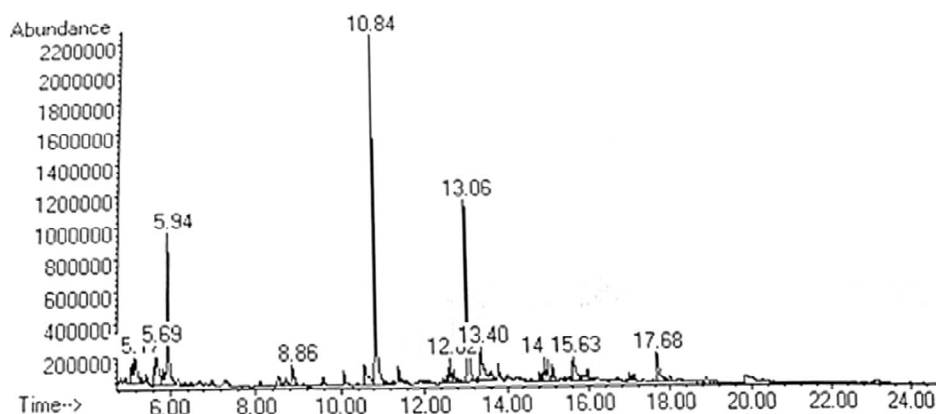


Fig. 1. GC-MS chromatogram of essential oil of marigold.

of essential oil of marigold flower collected from India. On the other hand, Iranian marigold contained β -caryophyllene (35.2%) as major component of essential oil of flower (Sefidkon *et al.* 2004). Similar differences in chemical compositions have also been reported in essential oils of other plants such as *Artemisia lavandulaefolia* (Zhang *et al.* 2012). The differences in chemical composition of essential oil of the same species could be attributed to seasonal and geographic factors (Huang *et al.* 2018). α -Terpinyl acetate, a natural monoterpene ester and a commercially

significant fragrance molecule. has also been reported as a major constituent of essential oils of *Stachys setifera* ssp. *iranica* (11.2%), *Chamaecyparis obtuse* (13.71%) and *Elettaria cardamomum* (46.12%) (Javidnia *et al.* 2003, Yang *et al.* 2007, Chowdhury and Kumar 2020). This compound possesses antioxidant and anti-amyloidogenic properties and also has disease amelioration effects in Alzheimer's disease (Chowdhury and Kumar 2020).

Table 1. Compounds identified in essential oil of marigold flowers by GC-MS analysis.

Sl. No.	Names of compounds	Molecular formula	Molecular weight	Retention time (min)	Peak area (%)
1	Propanoic acid, 2-bromo-2-methyl-, ethyl ester	C ₆ H ₁₁ BrO ₂	195.05	5.116	5.43
2	Cyclohexanol, 1-methyl-4-(1-methylethanyl)-, acetate	C ₁₂ H ₂₀ O ₂	196.28	5.693	5.33
3	Eucalyptol	C ₁₀ H ₁₈ O	154.25	5.943	14.27
4	<i>p</i> -Menth-1-en-8-ol	C ₁₀ H ₁₈ O	154.25	8.862	4.20
5	3-Cyclohexene-1-methanol, α , α ,4-trimethyl-, acetate	C ₁₂ H ₂₀ O ₂	196.29	10.844	31.67
6	Thymol	C ₁₀ H ₁₄ O	150.22	12.617	3.57
7	Phenol, 2,4-bis(1,1-dimethylethyl)-	C ₁₄ H ₂₂ O	206.32	13.058	14.35
8	Dodecanoic acid	C ₂₂ H ₂₄ O ₂	200.32	13.396	5.97
9	Octadecane	C ₁₈ H ₃₈	254.5	14.917	4.93
10	Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	228.37	15.626	5.58
11	<i>n</i> -Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	256.42	17.684	4.70

Two compounds, namely, phenol 2,4-bis (1,1-dimethylethyl)- (14.35%) and eucalyptol (14.27%) were found as abundantly occurring components of essential oil in the present study (Table 1). Phenol, 2,4-bis (1,1-dimethylethyl)- or 2,4-di-*tert*-butylphenol is a familiar secondary metabolite produced by a variety of organisms including bacteria, fungi, liverworts, diatom, plants and animals. It has antioxidant, anti-inflammatory, cytotoxic, insecticidal, antibacterial, antiviral, antifungal, phytotoxic and nematocidal activities (Zhao *et al.* 2020). Eucalyptol or 1,8-cineole, a monoterpene, a colorless liquid, having mint-like smell and made up 90% of eucalyptus oil (Boland *et al.* 1991), was found as a major constituent in essential oils of a variety of plant species including *Helichrysum gymnocephalum* (47.4%), *Rosmarinus officinalis* (43.7%) and *Artemisia lavandulaefolia* (35.60%) (Afoulous *et al.* 2011, Rašković *et al.* 2014, Huang *et al.* 2018). This compound has a number of biological activities including antibacterial, anti-inflammatory, antihypertensive and percutaneous penetration enhancer (Jiang *et al.* 2019). It has also been used in cosmetics, fragrances and as a flavoring agent (Bhowal and Gopal 2015).

Compounds present in moderate concentrations in essential oil of marigold included dodecanoic acid (5.97%), tetradecanoic acid (5.58%), octadecane (4.93%), propanoic acid, 2-bromo-2-methyl-, ethyl ester (5.43%), cyclohexanol, 1-methyl-4-(1-methylethanyl)-, acetate (5.33%), *n*-hexadecanoic acid (4.70%), *p*-menth-1-en-8-ol (4.20%) and thymol (3.57%) (Table 1). Dodecanoic acid or lauric acid, medium-chain white powdery solid saturated fatty acid and a major component of coconut oil (45-53%) possessed strong antibacterial, antifungal and antiviral potential (Dayrit 2014). Tetradecanoic acid or myristic acid is a long-chain saturated fatty acid

Table 2. Properties of compounds identified in essential oil of marigold flowers by GC-MS analysis.

Sl. No.	Names of compounds	Bioactivity	Reference
1	Propanoic acid, 2-bromo-2-methyl-, ethyl ester	Not reported	-
2	Cyclohexanol, 1-methyl-4-(1-methylethanyl)-, acetate	Not reported	-
3	Eucalyptol	Anti-inflammatory, antinociceptive, antihypertensive, antibacterial	Santos and Rao (2000),
4	<i>p</i> -Menth-1-en-8-ol	Antioxidant, antiproliferative, anti-inflammatory, antimicrobial	Sales <i>et al.</i> (2020)
5	3-Cyclohexene-1-methanol, α , α ,4-trimethyl-, acetate	Antioxidant, anti-amyloidogenic	Chowdhury and Kumar (2020)
6	Thymol	Antifungal, antibacterial, free radical scavenging, analgesic, anti-inflammatory, antioxidant, antispasmodic, antiseptic, antitumor	Meeran <i>et al.</i> (2017)
7	Phenol, 2,4-bis(1,1-dimethylethyl)-	antioxidant, anti-inflammatory, cytotoxic, insecticidal, antibacterial, antiviral, antifungal, phytotoxic and nematocidal	Zhao <i>et al.</i> (2020)
8	Dodecanoic acid	Antibacterial, antifungal and antiviral	Dayrit (2014)
9	Octadecane	Antibacterial, antifungal	Jasim <i>et al.</i> (2015), Barupal <i>et al.</i> (2019)
10	Tetradecanoic acid	Antifungal, antioxidant, nematocide	Mujeeb <i>et al.</i> (2014)
11	<i>n</i> -Hexadecanoic acid	Anti-inflammatory, cytotoxicity	Aparna <i>et al.</i> (2012), Ravi and Krishman (2017)

mostly found in milk and had antifungal, antioxidant, nematocide properties (Mujeeb *et al.* 2014). *n*-Hexadecanoic acid or palmitic acid is the most widespread saturated fatty acid in plants, animals as well as in microorganisms (Ferdosi *et al.* 2021, Javaid *et al.* 2021, Khan *et al.* 2021). It exhibited considerable cytotoxicity against human colorectal carcinoma cells (Ravi and Krishman 2017). *p*-Menth-1-en-8-ol or α -terpineol, a monoterpenoid alcohol, was known for its multiple biological properties such as antioxidant, anticarcinogenic anti-inflammatory, antimicrobial and anticonvulsant (Sales *et al.* 2020). Thymol, a monoterpene phenol, possessed a variety of pharmacological properties such as antifungal, antibacterial, free radical scavenging, analgesic, anti-inflammatory, antioxidant, antispasmodic, antiseptic and antitumor activities (Meeran *et al.* 2017). From the present study it may be concluded that essential oil of marigold flowers from Pakistan contains a variety of bioactive substance.

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(Manuscript received on 5 October, 2021; revised on 30 October, 2022)