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A Comparative LC/MS Analysis of Jordanian Lemon (Citrus limon): Peels, Pulp, Leaves, Branches, and Juice

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Abstract

Citrus limon (lemon) is important in pharmaceutical, restorative, and culinary (solid nourishment). It has uncommon properties and distinctive chemical compositions. The advantageous organic action of C. limon is decided by its tall substance of phenolic compounds, basically flavonoids (e.g., diosmin, hesperidin, limonin) and phenolic acids (e.g., ferulic, synaptic, p-hydroxybenzoic acids). The fundamental oil contains bioactive monoterpenoids such as D-limonene, Î²-pinene, and Î³-terpinene. As of late, deductively demonstrated restorative exercises of C. limon incorporate anti-inflammatory, antimicrobial, anticancer, and antiphrastic exercises. This considers the point of utilizing liquid chromatography-mass spectrometry (LC/MS) to examine the distinctive chemical components in different parts of Jordanian lemon. Lemon peels have the most extreme substance of hesperidin.

Keywords: *Jordan Lemon, Citrus Limon, Phenolic Compounds, Bioactivity, Phenolic Compounds, LC/MS.*

Introduction

The class Citrus has a place in the family Rutaceae and incorporates one of the most widely distributed natural products in the world Goetz P 2014 211 ,WH Talib 2010 1811. It speaks to an yearly production of roughly 124 million tons, of which the foremost imperative are oranges, Citrus sinensis L. (67 million tons); mandarins, Citrus reticulata L. (33 million tons); lemons, Citrus limon L.; and limes, Citrus aurantifolia L. (16 million tons) Papp N 2011;6:1459. The major makers of C. limon are India (3 million tons), China (2 million tons) and Mexico (2 million tons) Papp N 2011;6:1459

. New generation and preparing create a gigantic sum of waste, such as peels, seeds, and pulps, which speak to almost 50% of the crude natural product and are potential sources of added-value co-products. Even though a few have been valorized, other buildups are still arranged in landfills, causing environmental problems due to the molecules found in the peels, especially lipids, carbohydrates, and phenolic compounds, which also constitute a relevant loss due to their bioactive value. Valorizing these by-products increases the potential economic return while at the same time helping to preserve the environment IA Al-Ataby 2022 Bhatia H 2015202 ,Ba

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. Lemon is one of the most important crops in fruit production and is widely used in beverages, ice creams, and desserts, and as cooking ingredients, due to its tart flavor. Industrialization of lemon processing creates large quantities of by-products, which can be of interest to the food industry due to their high levels of nutrients and bioactive compounds (BCs), such as polyphenols, carotenoids, vitamins, essential oils (EOs), and dietary fiber, including pectin, cellulose, hemicellulose, and lignin Otang W 2016;46. Previous studies have shown that lemon peel (LP) contains higher levels of phenolic compounds and dietary fibers than the flesh Parhiz H 2015;223 Structurally, the LP consists of an outer layer, called the epicarp or flavedo, and an inner layer, called the mesocarp or albedo Kim J 2012;423. The flavedo contains high amounts of phenolic compounds, particularly flavonoids: hesperidin, diosmin, eriocitrin, and narirutin, whereas the albedo is rich in fibers, particularly pectin Bhavsar S.K 2007 303. The major components were limonene (57.7%), γ-terpinene (10.5%), and β-pinene (9. 3%). The most plant compounds in lemons:

-Citric corrosive. The foremost copious natural corrosive in lemons, citric corrosive may help anticipating kidney stones' arrangement.

â□¢ Hesperidin. This antioxidant may reinforce your blood vessels and avoid atherosclerosis â□□ the buildup of greasy stores (plaque) interior your courses Riaz A 2014;907 Abad-García B 2012 213 .â□¢ Diosmin. An antioxidant utilized in a few drugs that influence the circulatory framework, diosmin progresses muscle tone and diminishes inveterate irritation in your blood vessels García-Salas P 2013;869

- Eriocarpin. This antioxidant is found in lemon peel and juice García-Salas P 2013;869 Russo M 2015.307.

â□¢ D-limonene. D-limonene is the most common component of lemon fundamental oils and is found fundamentally within the peel and is dependable for lemons- particular smell. In confinement, it can diminish acid reflux and stomach reflux Talon M 2008:1 The lemon contains 3 main usable parts: pulp, pith, and peel (or some call it the skin). Pulps are vesicles that contain the juice of the lemon. It is a rich source of vitamin C, but there are also many other minerals and nutrients you didn't know it has, such as potassium, folate, vitamin B6, thiamine, copper, magnesium, and vitamin E. The pith contains fiber, it is a great source of antioxidants that can help reduce the risk of cancers and lower cholesterol. Lemon peels have an essence of their own. Lemon peels can contain at least 5 times the number of vitamins found in lemon juice. Other chemical compounds are incredibly useful to wellbeing, such as Silvestro Q40 and limonene, which offer assistance to battle cancer, polyphenol flavonoids which can help in clearing blood vessels that are blocked by cholesterol, and calcium which advances more robust bones and superior bone wellbeing. Lemon peels, which are utilizable foodstuffs, were analyzed for rough protein, phosphorus, calcium, copper, manganese, press, zinc, sodium, potassium, and a few chemical parameters in feedstuffs.

Materials and Methods

Plant Experimental Method

using Elute UHPLC coupled to a Bruker impact II QTOFMS.Chromatographic separation was performed using Bruker solo 2.0_C-18 UHPLC column (100 mm x 2.1 mm x 2.0 μm) at a flow rate of 0.51 mL/min and a column temperature of 40C. Solvents: (A) water with 0.1% methanol

and (B) Methanol injection volume 3 ul

Sample preparation:

the sample was dissolved with 200 ul DMSO with 2 ml methanol then centrifuged of each sample at 4000 rpm for 2.0 min, we Took 1.0 ml transferred it to an autosampler, and injected 3.0 ul (All standards used for identification of ms/z and the retention time)

2.2 Instrumentation and LC/MS Parameters

A Bruker Daltonik (Bremen, Germany) Impact II ESI-Q-TOF System equipped with Bruker Daltonik Elute UPLC system (Bremen, Germany) was used for screening compounds of interest. Standards for identification of m/z with high-resolution Bruker TOF MS and the exact retention time of each analyte after chromatographic separation were used. This instrument was operated using the Ion Source Apollo II ion Funnel electrospray source. The capillary voltage was 2500 V, the nebulizer gas was 2.0 bar, the dry gas (nitrogen) flow was 8 L/min, and the dry temperature was 200 ºC. The mass accuracy was < 1 ppm; the mass resolution was 50000 FSR (Full Sensitivity Resolution) and the TOF repetition rate was up to 20 kHz. using Elute UHPLC coupled to a Bruker impact II QTOFMS. Chromatographic separation was performed using Bruker solo 2.0_C- 18 UHPLC column (100 mm x 2.1 mm x 2.0 µm) at a flow rate of 0.51 mL/min and a column temperature of 40C. Solvents: (A) water with 0.05 % formic acid and (B) acetonitrile. Gradient: 0 – 27 min linear gradient from 5 % - 80 % B; 27 – 29 min 95 % B; 29.1 min 5 % B, total analysis time was 35 min on positive and 35 min on negative mode; injection volume was 3 uL.

Results and Discussion

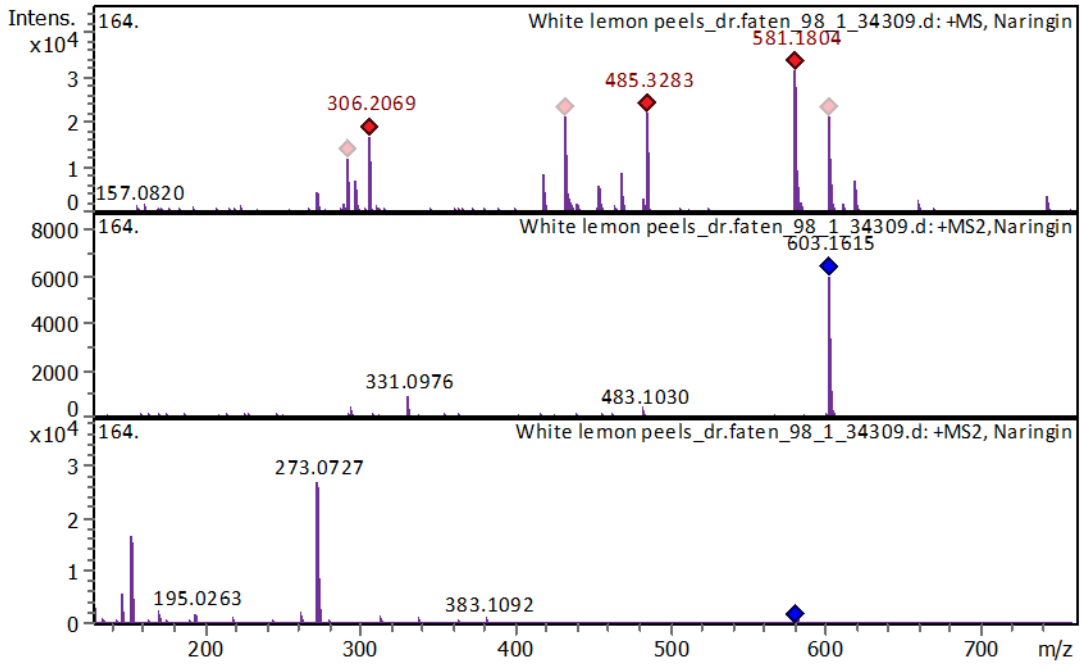
RT [min]	Compounds	Molecular Formula	White peel	yellow peel	Pulp	Branches	Leaves	Juice
3.37	Gallocatechin	$C_{15}H_{14}O_7$	1072	-	-	-	-	-
4.56	Catechin	$C_{15}H_{14}O_6$	2382	-	-	-	-	1014
6.75	Protocatechuic aldehyde	$C_7H_6O_3$	1130	-	-	-	-	-
7.31	Adenosine	$C_{10}H_{13}N_5O_4$	99618	186112	-	299632	255324	15610
7.4	Vanillic acid	$C_8H_8O_4$	23022	14662	-	11742	-	1092
10.71	4-Methylumbelliferone	$C_{10}H_8O_3$	882	-	-	-	-	-
10.84	o-Coumaric acid	$C_9H_8O_3$	23434	10478		65250	88154	71664
11.1	Ferulic acid	$C_{10}H_{10}O_4$	99462	33858		23432	18242	1782

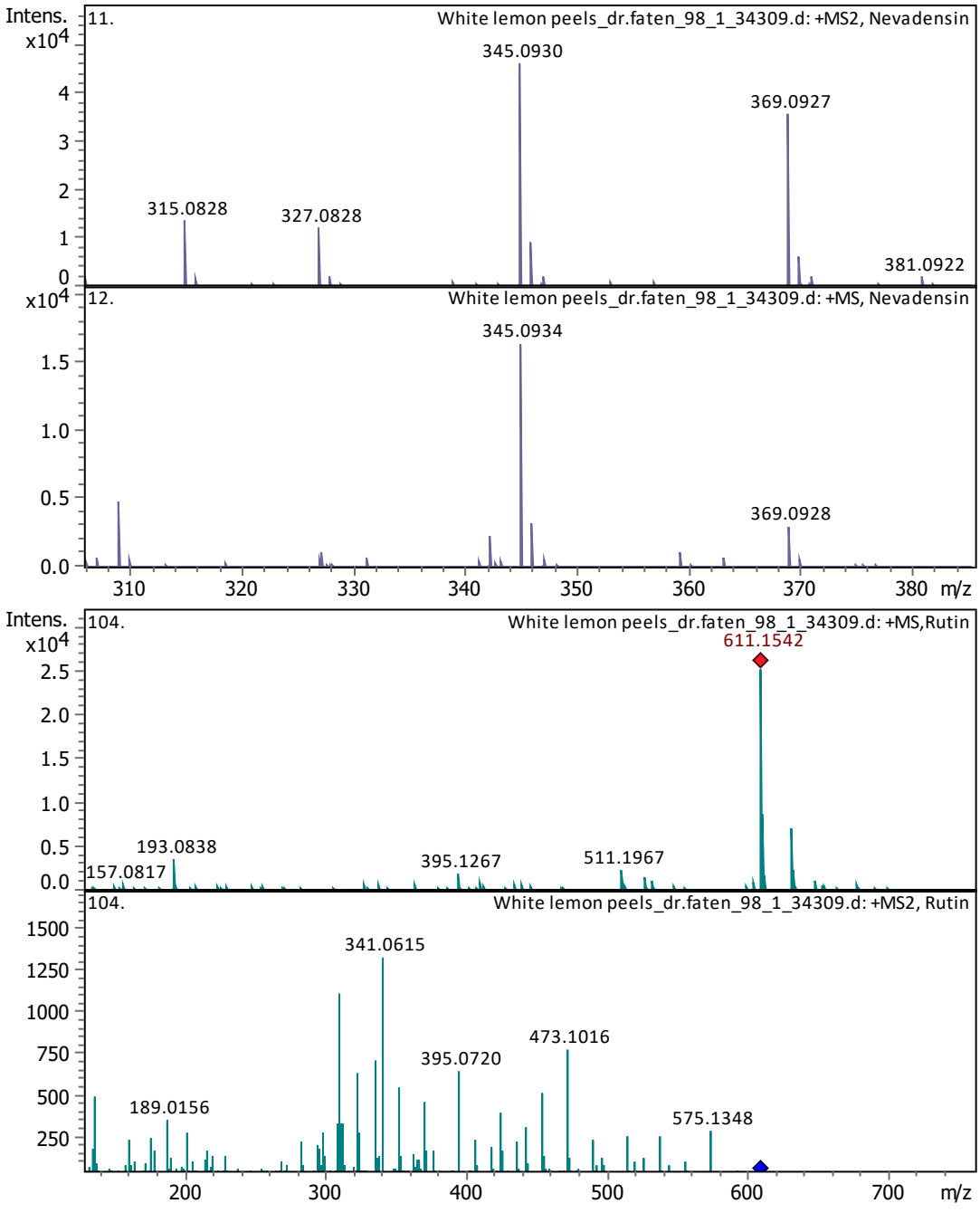
11.11	5-Glc tricin (NMR)	$C_{23}H_{24}O_{12}$	268	1360				
12.58	Rutin	$C_{27}H_{30}O_{16}$	25552	53596		117592		8292
12.64	Isoorientin	$C_{21}H_{20}O_{11}$	8060	6174		18052	49196	
13.02	Acacetin-5-O-xyloside	$C_{21}H_{20}O_9$	11980	8944		10048	5512	
13.02	Naringin	$C_{27}H_{32}O_{14}$	192		660	2884		948
13.04	Vitexin	$C_{21}H_{20}O_{10}$	21044	13080		49120	175140	
13.59	Gardenin A	$C_{21}H_{22}O_9$	15684	5168		4860	3376	
13.59	Nevadensin	$C_{18}H_{16}O_7$	22490			22050		17270
16.69	Hesperidin	$C_{28}H_{34}O_{15}$	1998958	1274278	10696	2049250		58022
19.45	7,4'-Dimethoxy-3-hydroxyflavone	$C_{17}H_{14}O_5$	16480					
22.84	3-Oxocostusic acid	$C_{15}H_{20}O_3$	444					1592
27.56	Rosmanic acid	$C_{18}H_{16}O_8$		1098				
29.81	7,3'-Dimethoxy-5,6,4'-trihydroxy isoflavone	$C_{17}H_{14}O_5$		4798		5522	1534	
19.45	Quercetin	$C_{15}H_{10}O_7$		6948		15084	39234	
12.98	Ascorbic acid	$C_6H_8O_6$		5536	1240			18804
11.41	4-hydroxy cumarin	$C_9H_6O_3$		85110		29874	21018	108440

8.69	Palmitoleic acid	$C_{16}H_{30}O_2$		30772			286396	
28.58	Temephos	$C_{16}H_{20}O_6P_2S_3$						138916
13.02	stigmasterol	$C_{29}H_{48}O$		1318				
13.04	Gallic Acid Ethyl Ester	$C_9H_{10}O_5$				15450		1884
13.59	Anisic acid	$C_8H_8O_3$				18252		
13.59	Caffeic Acid	$C_9H_8O_4$				9602		2498
14.99	Scopoletin	$C_{10}H_8O_4$						992

Table: Components of Lemon Peels, Pulp, Branches, Leaves And Juice

Done by researcher




Intens.
x10^4
11.
White lemon peels_dr.faten_98_1_34309.d: +MS2, Nevadensin
345.0930
369.0927
315.0828
327.0828
381.0922
12.
White lemon peels_dr.faten_98_1_34309.d: +MS, Nevadensin
345.0934
369.0928
m/z
104.
White lemon peels_dr.faten_98_1_34309.d: +MS,Rutin
611.1542
193.0838
157.0817
395.1267
511.1967
104.
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473.1016
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575.1348

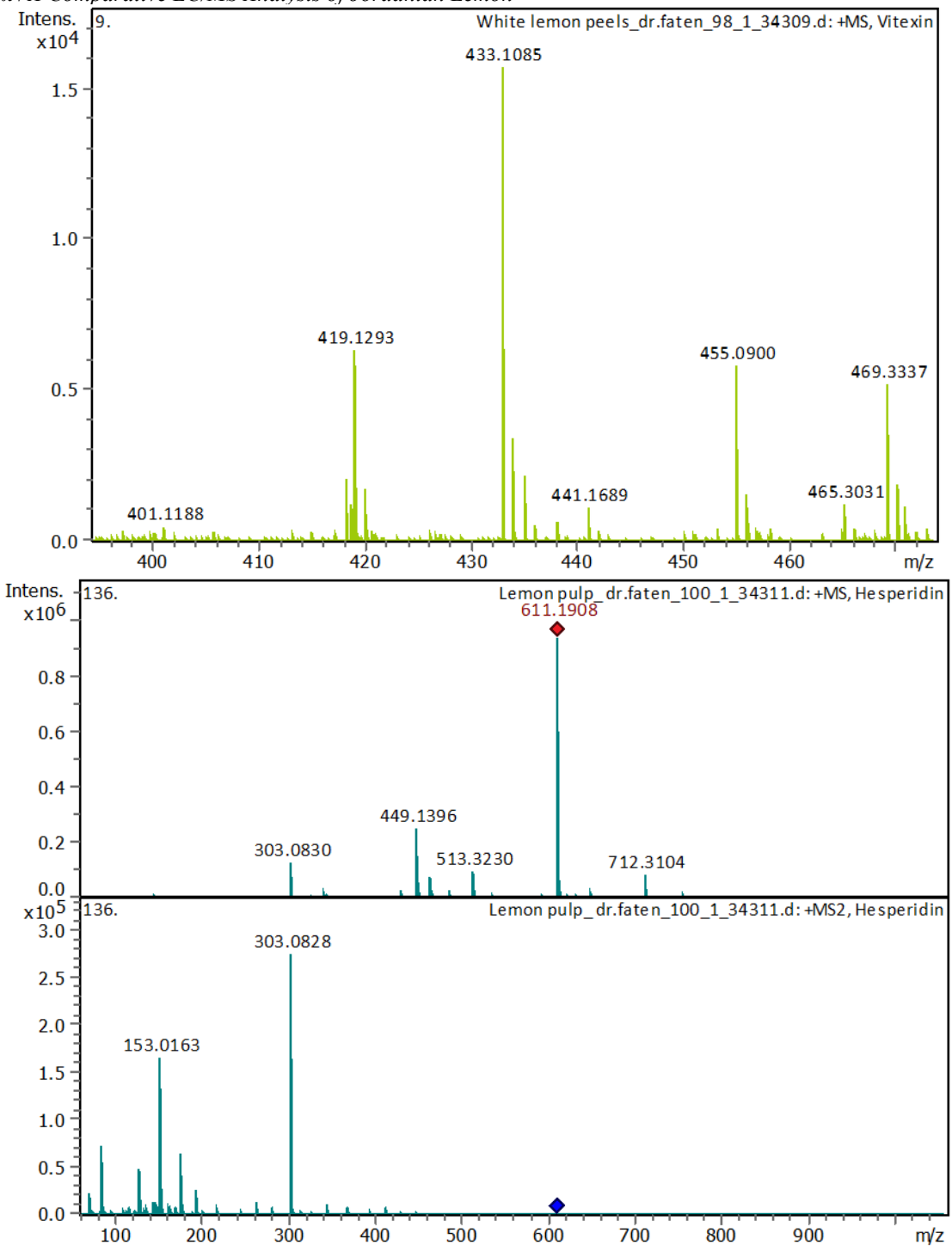

Figure 1: Mass Spectra of the Most Common Compounds Found in Limon

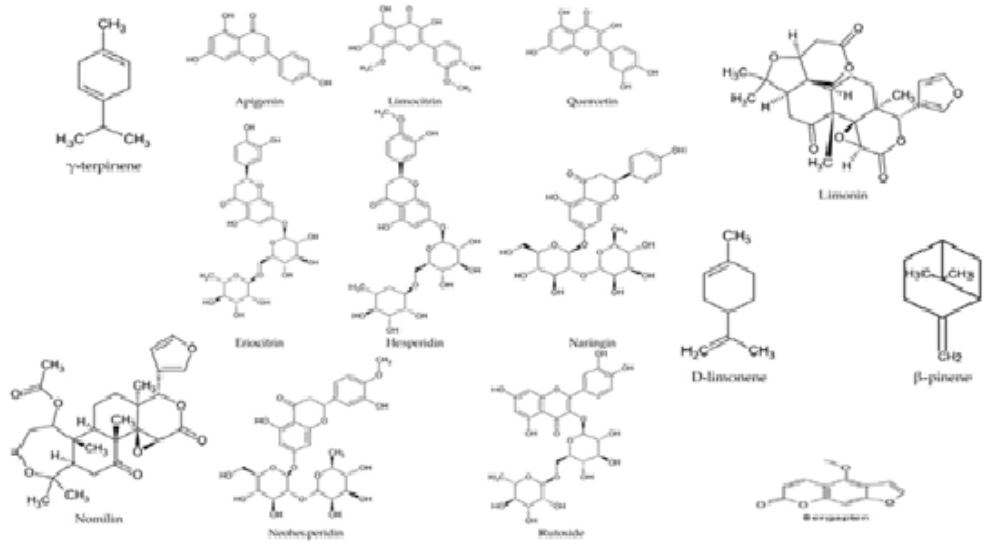

Figure 2:Structures of organic compounds

Discussion

Phenol and phenolic acid: Phenolic acids like ascorbic acid are present in all parts of limon.Jing L. 2015,Citrus Page 2019. The stilbinoid Astringin is present in the yellow peel only.Hagerty M 1932,Herbert S 1943.The coumarin is common in both rind and pulp of Citrus limon Swingle W.T,1943 ,Millet F 2014, There are no fatty acids and lipids are present in all parts of the citrus lemon.European Directorate for the Quality of Medicines 2017,The United States Pharmacological Convention 2009.The organic acid citric acid is present in both rind and pulp, and pimelic acid is present only in rind. Phenyl propanoids like Cinnamic acid is common in the Pulp and rind of Citrus lemon fruitsMinistry of Health and Faily Welfare 2017 Trans para coumaric acid is present only in Pulp, and Caffeic acid glucoside and Rosmarinus acid are present only in Rind.Agence 1998

Conclusion

The presented review proves that C. Limon is a beautiful object of different scientific studies. The C. lemon fruit is a raw material that can be used in different forms, e.g., extracts, juice and essential oil. The rich chemical composition of this species determines a wide range of its biological activity and its being recommended for use in phytopharmacology. The studies have focused on the essential oil and its main active compound—D-limonene. Extracts from C. lemon fruits are rich in flavonoids such as naringenin and hesperetin. Current pharmacological studies have confirmed the health-promoting activities of C. limon, especially its anti-cancer and antioxidant properties. C. limon also finds increasing application in cosmetology and food

production.

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