

Chemistry of the Main Component of Essential Oil of *Litsea cubeba* and Its Derivatives

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Abstract

The tree of *Litsea cubeba* is widely spread in China, Indonesia and other part of Southeast Asia. The essential oil of *Litsea cubeba* (EOLC) is obtained by steam distillation from the pepper-like fruits tree *Litsea cubeba*. The EOLC consists of about 29 active compounds. Among them, citral is the main component; the content of citral is nearly 80% of the EOLC. Due to the special function group, citral is easy to react with many chemicals. Thus, EOLC is usually applied as starting material to carry out aldol condensation, reduction, and six-member ring forming reaction. The EOLC is extensively employed to synthesis of geranal nitriles, pseudonoe, ionone, methyl ionone, Vitamin E and Vitamin A. These products are broadly applied in the fields of fragrance, perfume, medicine and so on. This paper presents comprehensive utilization of EOLC as raw materials to synthesize many active chemicals.

Keywords

Litsea cubeba, Essential Oil, Citral, Synthesis

1. Introduction

In the recent years, the utilization of essential oil both in daily life and industry has increased exponentially as use of one or more components from plants like *pinus* (Packer et al., 1999; Ayrlmis et al., 2009), *Litsea cubeba*, *Cinnamomum camphora* (Linn.) Presl (Stubbs et al., 2004; Qian et al., 2012) and so on. Essential oil is extensively used in medicine, nutrition, flavoring, beverages, dyeing, repellents, fragrances, cosmetics (Djeridane et al., 2006). For many years, utilization of essential oil has played increasingly important role in the strategy of chemistry industry field (Isman, 2000). As a part of this preoccupation, the researches for high efficient, green,

and high valued components from active plants are of vital concern. In addition, compared to the active components obtained by solvent extraction and synthesis, essential oils are commonly obtained by water vapor distillation, thus essential oils are eco-friendly, low-toxicity. Furthermore, most of essential oils have excellent effects on benefiting human beings in food, medicine, perfume and fragrance (Bakkali et al., 2008).

The essential oil of *Litsea cubeba* is obtained by steam distillation from the pepper-like fruits (Figure 1) *Litsea cubeba* (Figure 2). The tree is widely spread in China, Indonesia and other part of Southeast Asia, where it occurs in mountainous region. In ancient China, the EOLC is employed as medicine for headache, fatigue, muscle pain and depression (Chen et al., 2013). Nowadays, The EOLC is extensively employed to prepare geranyl nitriles, pseudonoe, ionone, methyl ionone, Vitamin E and Vitamin A. Herein, we report the chemistry of the EOLC and main components of EOLC as starting materials in chemistry and industry.

2. Ease of Use

2.1. Chemistry of the EOLC

The EOLC had been analyzed by several researchers (Ye, 1992; Cheng, 1995; Liu et al., 2001; Wang et al., 2002). Recently, it reported that the essential oil of *Litsea cubeba* contained 29 compounds (see Table 1) (Hu et al., 2011), which mainly contains terpenes, lipids and fatty acids. On account of one of the double bonds binding with four different groups, citral has two geometric isomers, which are the E isomer (geranial) and the Z isomer (neral) (Scheme 1). The content of citral in EOLC was as high as 70%. The content of limonene was 10% and the content of other components was 20% approximately (Table 1). The experiments details as follows:

2.1.1. Materials and Chemicals

Litsea cubeba Fruits were obtained from local *Litsea cubeba* trees within 6 h stored at -20°C in freezer.

Chemicals and Reagents. Standards were purchased from Sigma-Aldrich Company (American). The solvents were analytical grade and without treatment before use.

2.1.2. Extracting EOLC

EOLC were obtained through vapor distillation of the fruits of *Litsea cubeba*. General procedure was as follows: 5 g fruits of *Litsea cubeba* were added into 250 ml round bottom flask with 100 ml water and using vapor distillation equipment for vapor supplying, collecting the oil and dried with sodium sulfate and concentrated by vacuum rotary evaporator to 2 ml. Followed by further concentration to 0.2 ml using a flow of nitrogen.

2.1.3. GC, GC-MS Analysis

GC analysis was performed on an Agilent 6890. The gas chromatography was equipped with a FID. The separation was performed using a nonpolar fused silica capillary column HP-5 (5% Phenyl Methyl Siloxane, Agilent 19091 J-413), 30.0 m \times 0.32 mm coated with 0.25 μm film thickness. Carrier gas (Nitrogen) at flow rate of 1 ml/min. Column temperature program was 50°C for 5 min and then programmed to 85°C for 2 min at $5^{\circ}\text{C}/\text{min}$, then increased to 280°C for 10 min at $3^{\circ}\text{C}/\text{min}$.

GC-MS analysis were performed on a HP6890 gas chromatograph equipped with a HP 5973 mass selective detector using a 30 m \times 0.25 mm id., 0.25 μm film thickness HP-5 capillary column (5% Phenyl Methyl Siloxane, Agilent 19091 J-413) with helium as carrier gas. Column temperature program was 50°C for 5 min and then programmed to 85°C for 2 min at $5^{\circ}\text{C}/\text{min}$, then increased to 280°C for 10 min at $3^{\circ}\text{C}/\text{min}$.

Volatile compounds were identified by comparing their mass spectra with the mass spectra from MS database (NIST 05, WILEY 7). When available, MS identifications were confirmed by comparing GC retention times of the analysts with those from pure standards. The identification was confirmed by using retention indices (RI) of the value compared with those reported in the literature (Jordan, M. J. et al., 2002). Linear retention indices of the compounds were calculated using a series of n-alkanes ($\text{C}_7\text{-C}_{30}$, Sigma-Aldrich, America) injected in the same conditions. When standard chemicals were not available, tentative identification was carried out by matching the mass spectra. The results were given in Table 1.

2.2 Synthesis of Geranyl Nitrile

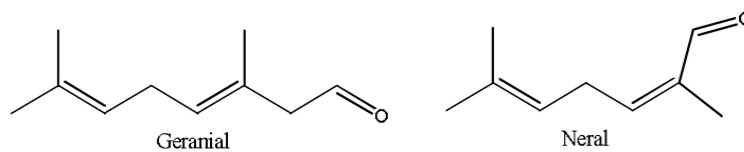
Geranyl nitrile with lemony-fresh odor similar to citral (Calkin and Jellinek, 1994) is widely used as fragrance



Figure 1. Fruits of *Litsea cubeba*.



Figure 2. The tree of the *Listea cubeba*.



Scheme 1. The two geometric isomers of citral.

ingredients and incorporated into a variety of consumer product types, including soaps, shampoos, cosmetics, perfumes, detergents and insect repellents (Potter et al., 2001; Arcander, 1969). The geranyl nitrile is appre-

Table 1. Chemical composition (%) of EOLC (Hu, 2011).

Components	RI ^a	Peak area (%)	Identification method
α -Pinene	932	0.58	GC-MS ^b
β -Phellandrene	946	trace	GC-MS
Camphene	956	0.48	GC-MS
β -Pinene	978	0.19	GC-MS
6-Methyl-5-en-2-one	988	trace	GC-MS, RI ^c
<i>p</i> -Cymene	993	1.22	GC-MS
Cineole	1032	trace	GC-MS, RI
<i>d</i> -Limonene	1041	8.52	GC-MS, RI ^d
γ -Terpinene	1045	0.58	GC-MS, RI
Linalool	1104	1.36	GC-MS, RI
<i>cis</i> -Limonene oxide	1132	0.85	GC-MS, RI
Thujanol	1139	0.98	GC-MS, RI
(<i>E</i>)- <i>p</i> -Menth-2-en-ol	1140	0.74	GC-MS, RI
Citronellal	1152	0.94	GC-MS, RI
Borneol	1165	0.2	GC-MS, RT
Terpinene-4-ol	1178	0.27	GC-MS
α -Terpineneol	1196	0.63	GC-MS
citronellol	1217	0.48	GC-MS
β -Citral	1248	26.15	GC-MS, RT
Nerol	1257	0.98	GC-MS
α -Citral	1265	33.16	GC-MS, RT
Bornyl acetate	1282	3.98	GC-MS, RI
Eugenol	1358	1.19	GC-MS, RI
Caryophyllene	1454	3.47	GC-MS, RI
Ethyl myristate	1791	1.87	GC-MS
Palmitic acid	1971	1.16	GC-MS
Ethyl palmitate	1993	2.25	GC-MS
Methyl oleate	2094	0.57	GC-MS
Linoleic acid ethyl ester	2144	1.32	GC-MS
Ethyl oleate	2176	1.72	GC-MS

^aRetention Index; ^bIdentified by good match of mass spectrometer; ^cIdentified by retention index and compared with those reported in the literature (Qin, 2008); ^dIdentified by retention time of standard compounds.

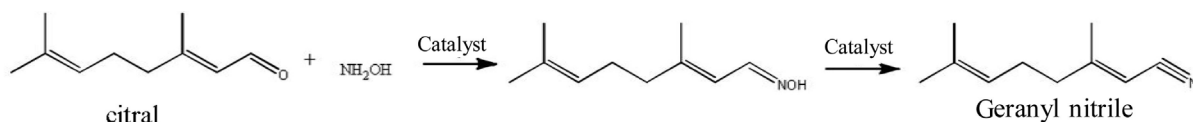
ciated for its comparative stability under mildly alkaline conditions, which makes it particularly suitable to applications in soaps, detergents.

Zheng reported citral directly reacting with ammonia in the presence of active copper and molecular sieve as catalyst. The yield could reach 90% (Scheme 2). However the reaction demonstrated some disadvantages: Firstly, the reaction endured so high temperature that the reaction led to more energy consuming; Secondly, this reaction had many side reactions, thus the product was difficult for purification. In 2001, Nong adopted tetrabutylammonium bromide as transfer phase catalyst, and acetic anhydride as dehydrant in mild condition to synthesis of geranyl nitrile; and the yield was 91% (Nong et al., 2001). Zhou carried out the reaction with hydroxyl amine sulfate. This procedure was more facile and convenient. And yields were good to excellent. However, excess amount of citral was employed (Zhou et al., 2005).

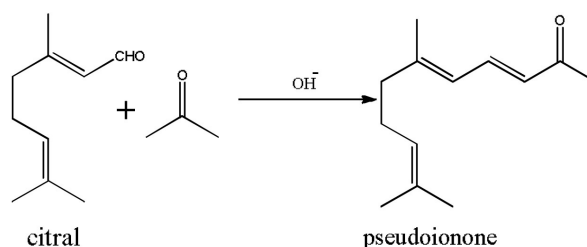
2.3 Synthesis of Pseudoionone

Pseudoionone is a key intermediate for preparation of ionone. Usually, pseudoionone is applied in fragrance, food additives and perfume field (Rajuet et al., 2011).

Pseudoionone is prepared by citral condensing with acetone in the presence of catalysts (Scheme 3) basically.



Scheme 2. Synthesis of geranyl nitrile with ammonia.



Scheme 3. Preparation of pseudoionone using citral and acetone as starting materials.

This reaction is simple and facile to carry out. Researchers were focusing on exploring the catalysts. Zhou reported the aldol condensation between citral and acetone. The reaction was performed by using catalytic amount of sodium hydroxide or potassium hydroxide (2%) in the presence of 6 equivalents of acetone; moderate to good yields were obtained (Zhou et al., 2009). Catalyst was crucial for the reactions, and many catalysts were screened and modified for improving the reactions, such as: EtONa/EtOH (Wang, 1990), KOH/MeOH (Wang and Jiang, 1993) and so on. The yields of the reactions could get 86%, and purity could get 97%. However, there were still many side reactions such as citral self-condensation, acetone self-condensation and so on occurring in the reaction.

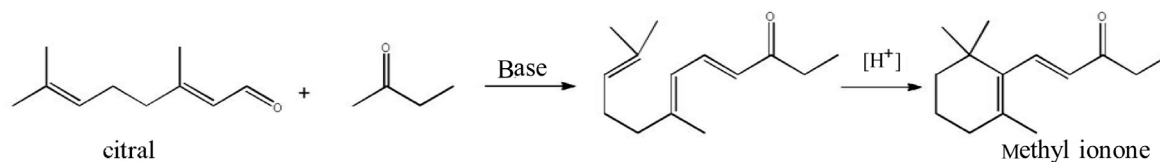
2.4. Synthesis of Methyl Ionone

Methyl ionone known as high-grade fragrance with fresh odor is mainly used in fragrance and food additives. In 1980, Guo described the synthesis of methyl ionone use KOH and methanol as condensation agent, and the yield was 89%. Meanwhile, using phosphoric acid as cyclization agent, and the yield was 70% (Guo et al., 1980). In 2002, Lin investigated macroporous resin as catalysts for cyclization of the pseudomethylionone to produce methylionone (Lin and Liang, 2002; Tang, 2011). The methyl ionone could be prepared by aldol condensation and ring-closed reaction (Scheme 4). The typical procedures were as follows: methyl acetone in 250 ml flask added 5% NaOH 30 ml with stirring, then dropped EOLC at 35°C - 40°C for a few hours in the presence of catalysts. In 2003, Huang reported that citral and 2-butyl acetone as starting materials in the presence of quaternary ammonium hydroxide and solid super acid $\text{TiO}_2/\text{SO}_4^{2-}$ as catalyst. Yields were 90% - 92%. However, the dimethyl benzene as solvent was difficult to remove off from the products. In 2004, Lin developed a new method: the anion exchange resin as catalyst, the yield was quantitative, but the large amount of catalyst was consumed (Lin 2004).

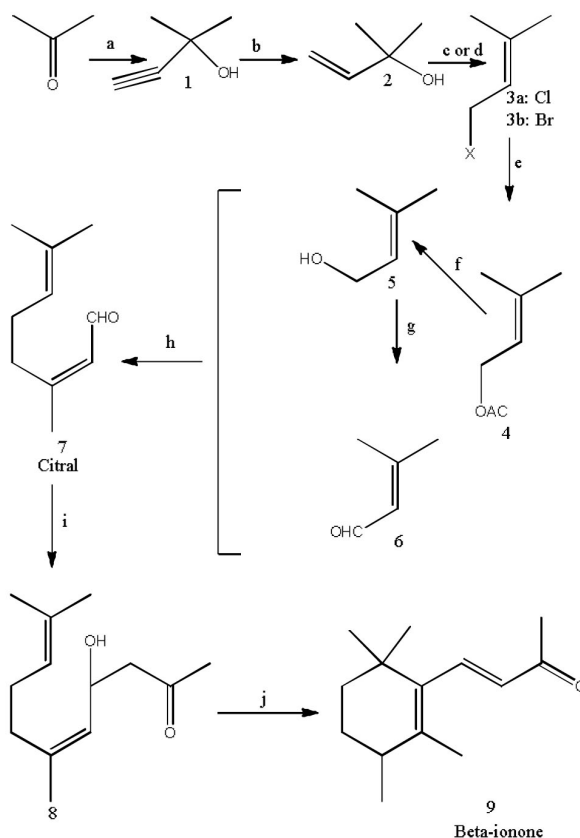
2.5. Synthesis of β -Ionone

β -ionone, an important contributor to fragrance in many flowers (Kaiser and Lamparsky, 1980; Cooper et al., 2003), is an abundant source of useful aroma compounds existing in a variety of essential oils with various application in fragrance, perfume industry. In addition, β -ionone is key intermediate to prepare vitamin E, vitamin A and extensively used in cosmetics (Kanei et al., 1999), food flavor and inhibitor (Yu et al., 1995; Plotto et al., 2006).

Citral condensing with acetone under base condition is the typical method of synthesis of β -ionone, then through ring-closure reaction forming β -ionone in the presence of catalyst (Scheme 5) (Hertel et al., 1986). During the whole reaction, selecting the excellent catalyst is vital for the whole reactions. Hence, many researchers had been working on the selecting and applying catalysts such as sodium hydride, $\text{KF}/\text{Al}_2\text{O}_3$, LiOH (Lu and Yao, 2007; Zhang et al., 2010), phosphoric acid. Transfer phase catalyst $\text{Bu}_4\text{N}^+\text{Br}^-$, $\text{Et}_3\text{N}^+\text{CH}_2\text{PHCl}^-$ (Liu et al., 2000). The yields varied from mediate to good.



Scheme 4. Synthesis of Methyl ionone.



Scheme 5. Typical method of synthesis of β -ionone.

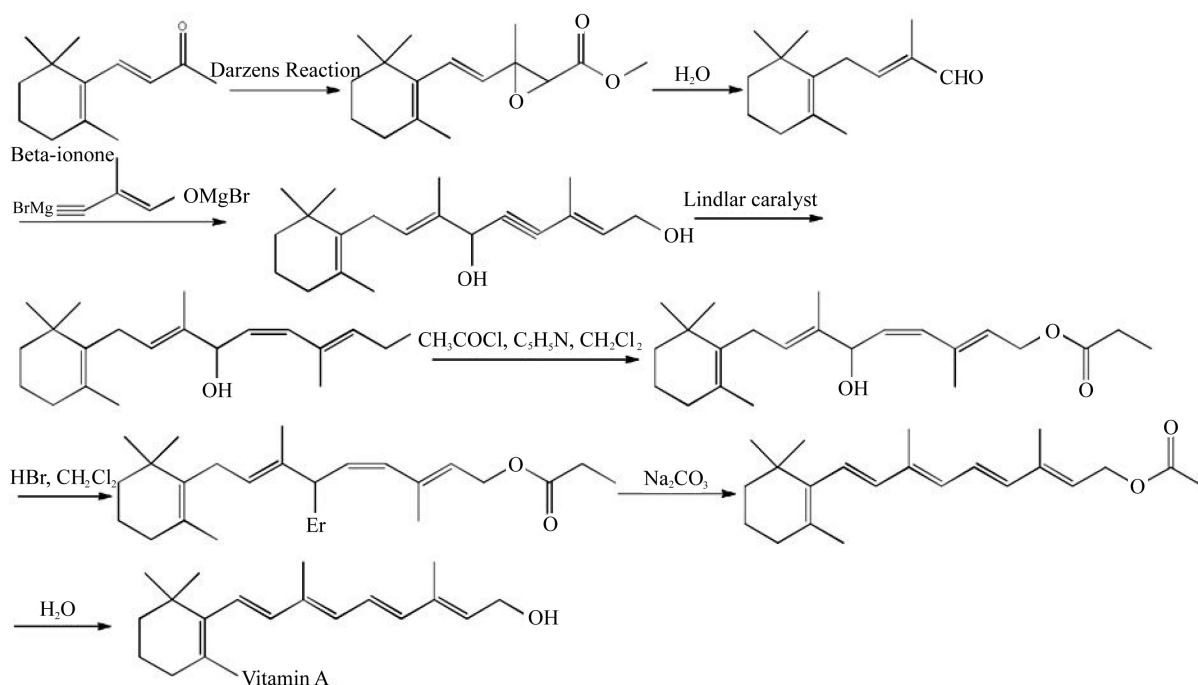
2.6. Synthesis of Vitamin A

Vitamin A is a group of unsaturated nutritional organic compounds, which includes retinol, retinal, retinoic acid, and several provitamin A carotenoids (Damodaran and Parkin, 2008). Vitamin A is a vital medicine, which is widely used for nyctalopia, eye and skin dryness. It is important for growth and development for the maintenance of the immune system and vision (Tanumihardjo, 2011).

Vitamin A was first synthesized in 1947 by two Dutch chemists. Then, scientists and industry managed to commercialize Vitamin A (Robeson et al., 1955). Vitamin A has a special structure: β -ionone ring to which an isoprenoid chain attached. In 1947, the Roche Company commercialized the product using beta-ionone as starting materials through Dazens reaction, Grignard reaction, hydrogenation acetylation, bromination, elimination (Scheme 6). However, 50 more reagents were employed and many reaction units were employed. Years later, BASF Company invented a new route that beta-ionone as starting materials underwent ethynylation, reduction and elimination. This synthetic pathway was simple and economical. Meanwhile the yield was high. However, the drawbacks were secondary key intermediates C_5 aldehyde difficult to synthesizing.

2.7. Synthesis of Vitamin E

Vitamin E is the most important industrial fat-soluble antioxidant, which includes tocopherols and tocotrienols,



Scheme 6. Synthesis of Vitamin A.

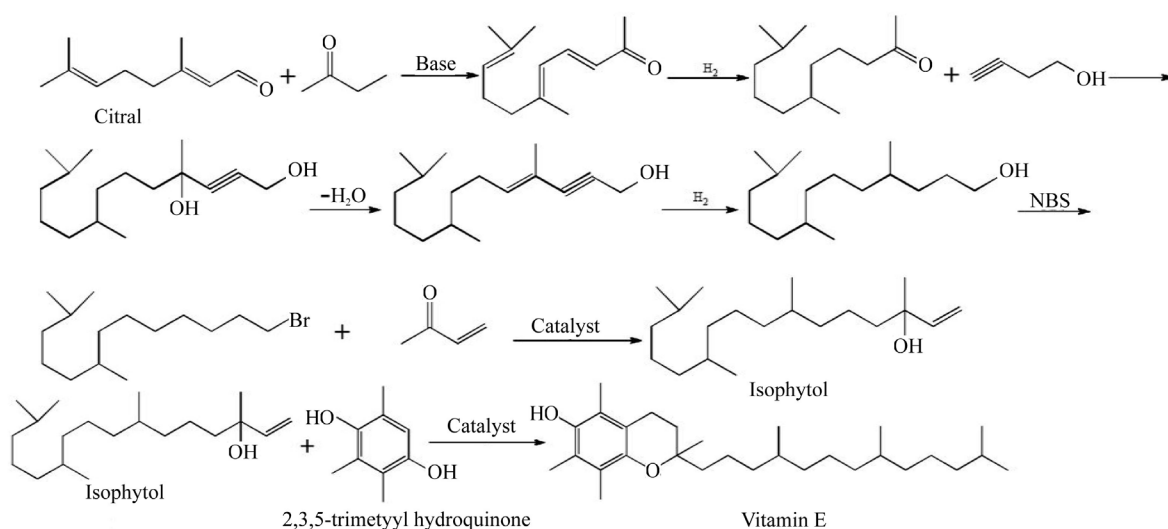
extensively applied as antioxidant, anti-cancer drugs, free radical scavenger (Bowry et al., 1995). Vitamin E could improve human physical immunity, and used as animal feeds (Bonrath et al., 2007).

Vitamin E, generally, is synthesized by two building blocks through the Friedel-Crafts alkylation reaction. One building block is 2,3,5-trimethyl hydroquinone (TMHQ), and the other building block is isophytol (Scheme 7). And the mechanism of the reaction is enol under the catalyst of AlCl_3 forming carbocations binding with TMHQ. To date, there are only four companies producing Vitamin E in large scale. Isophytol is a key material for the synthesis of vitamin E. Usually, isophytol was synthesized by using citral or pseudoionone as starting materials, through serials of reactions (Sato et al., 1963). And the study to date was focused on catalyst selecting and modification. Many solid acids were applied to replace the liquid acid. Qian used phosphomolybdic acid as catalyst obtained good yield. The purity could get 88% (Qian et al., 1999). Makoto employed scandium (III) trifluoromethanesulfonate as an efficient catalyst (Matsui et al., 1995). Bulychev employed amine as catalyst (Bulychev and Kartsev, 1999).

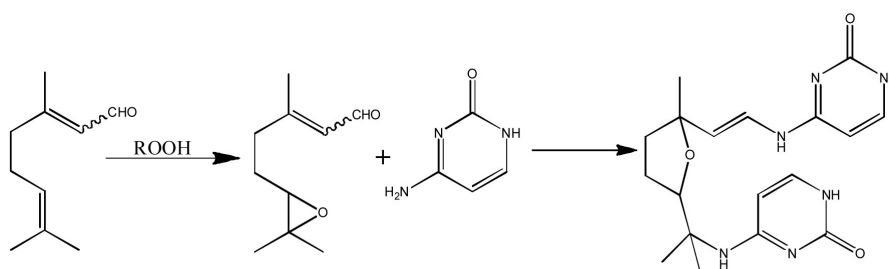
2.8. Synthesis of Nitrogen Derivatives

The citral could condensation with amines and amino acids. Saddiq used citral reacted with cytosine, this product showed the good antibacterial and antifungal activity, especially on staphylococcus aureus (Saddiq and Khayyat, 2010). And the synthesis route as follow (Scheme 8).

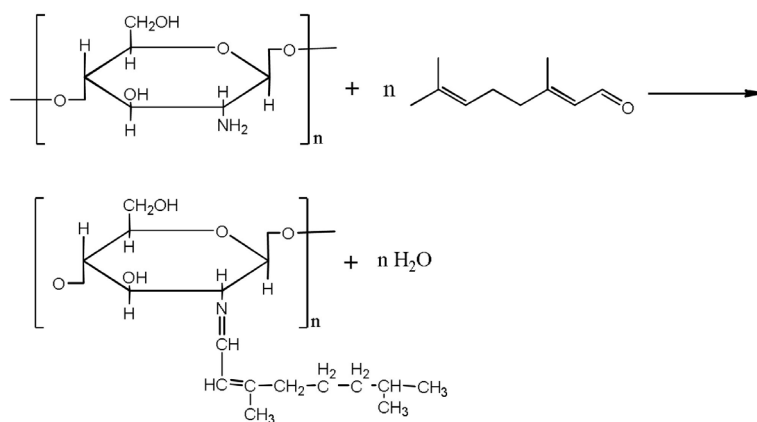
Chitosan, a biocompatible, biodegradable, non-toxic polymer, is prepared from chitin, which is the second most naturally occurring biopolymer after cellulose. The Schiff base of chitosan was synthesized by the reaction of chitosan with citral under high-intensity ultrasound (Scheme 9). The effect of the molar ratio of chitosan to citral, reaction time, and temperature on the yield has been investigated. The optimal conditions were a temperature of 50°C , a molar ratio of chitosan to citral of 1:6, and a reaction time of 10 h. The maximum yield achieved was 86.4% under optimum conditions. The structure of the schiff base was characterized by FTIR spectroscopy, elemental analysis, and X-ray diffraction studies. The strong peaks at 1648.3 and 1610.6 cm^{-1} are due to C-N bond and C-C bond stretching vibrations. The results confirmed that amino groups on chitosan reacted with citral to form the Schiff base. The antimicrobial activities of chitosan and Schiff base of chitosan were investigated against *Escherichia coli*, *Staphylococcus aureus*, and *Aspergillus niger*. The results indicate that the antimicrobial activity of the Schiff base increases with an increase in the concentration. It was also found that the



Scheme 7. Synthesis of Vitamin E.



Scheme 8. Synthesis of nitrogen derivatives with nitrals as starting materials.



Scheme 9. Synthesis of chitosan derivatives.

antimicrobial activity of the Schiff base was stronger than that of chitosan (Jin, 2009).

3. Conclusion

With the information to date, it has shown that the EOLC could be employed to synthesis such as pseudoione, iodone, methyl iodone, Vitamin E, Vitamin A and so on. These products were widely applied in perfume, fragrance, food flavor, anti-inflammatory, anti-bacterial, and antifungal medicine. Some products are very popular

in daily life. Some new nitrogen derivatives from EOLC could also discover some new functions.

As shown in this review, despite of much research works and application, improvements had been done on the chemical utilization of EOLC. However, most of the research work was focused on modifying and selecting the catalysts for improving yields or simplifying the reaction procedures; exploiting new EOLC based compounds to expand the utilization of the EOLC needs much more work. On the other hand, besides the citral, the EOLC has many other components such as lipids and fatty acids; these compounds should be well studied.

Acknowledgements

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