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Synthesis of Perfumery Esters using Green Catalyst.

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Abstracts -
Esters are valuable chemicals used in the perfumery industry. Several synthetic routes are followed to synthesize esters but esterification is a widely used procedure however these methods have the limitation of using corrosive acid as catalyst. when such effluents are discharged in the environment invites threats to aquatic life.
The current work utilizes a solid acid catalyst, Amberlyst 15 as a green catalyst to synthesis commercially important perfumery esters. The present paper describes the synthesis of esters like L-Menthyl acetate, Cis-3-Hexenol acetate, Terpinyl acetate, Citranellol acetate, Geraniol acetate, Linalool acetate, 2-phenyl ethyl acetate respectively. The percentage conversion of the product was found to be more than 90 % and finally, the purity was checked by gas chromatography. The proposed method is selective energy efficient and the catalyst used can be recyclable offers a green prospect that helps to reduce environmental pollution.

Keywords-
L-Menthol, Cis-3-Hexanol, alpha Terpineol, Citranellol, Geraniol, Amberlyst 15, Acetic Acid, Acetic Anhydride.

Introduction-

Esterification is one of the important reactions which produces organic esters having applications or act as intermediates in the synthesis of perfumes. These esters can be synthesized via different routes as solvolytic, condensation, free radical processes, PTC, etc. [1]. But among all, only a few of the processes meet the specification of perfumery. The most widely used method is the reaction of acid and alcohol to prepare corresponding esters in presence of solid heterogeneous catalyst [2].
Generally liquid phase homogenous catalysts such as *p*-toluene sulphonic acid, methane sulphonic acid, phosphoric acid, hydrochloric acid, *etc*. Have been used industrially for such processes. But the homogenous acids cause serious environmental damages creating acidic waste water, also posing some problems like difficulty in handling, imparting odor, difficulty in recovering the catalyst after reaction, etc.
The above limitation was over come by the use of heterogeneous catalyst having the advantage as recovery from the reaction mass, high reusability, required in small quantity, environmentally friendly process. [3-4].
The main aim of our work is to use environmentally green catalysts which will prevent pollution and helps to reduce pollution impacts on the environment. The experimental setup is a very simple four-neck round bottom flask with Dean and Stark set up under heating, stirring and reflux mode condenser. A respective Alcohol, Acetic Acid, Amberlyst 15 as a catalyst, reaction mass heat to reflux and continuous removal of the aqueous

layer from side arm of Dean and stark. When water separation slows down, cool reaction mass and remove the sample and analyzed by Gas Chromatography instrument and accordingly unreacted alcohol add Acetic Anhydride and reflux reaction mass for another one hour. Separate the solid resin catalyst and mass taken for fractional distillation under vacuum (5-10). The perfumer by only smelling tell the quality of products and it will pass perfumery test in the market or not. Thus, the appointment of a perfumer is a very important task in the perfumery industry.

Experimental setup-

Experimental process-

The experimental setup consists of a 250 mL round bottom flask with Alcohol [1 Mole], acetic acid [1.3 Moles], Amberlyst 15 [10%], charged in the reaction vessel and heat to reflux. The continuous removal of water from the dean and stark side arm and recycle of top organic layer again in the reaction vessel. When water separation slows down, cool the reaction mass and added through addition funnel 2% of Acetic Anhydride and again refluxed for one hour. The reaction mass was cooled to separate solid catalyst which was recycled and the product separated by fractional distilled under high vacuum to get high purity. As nearly all perfumery products are high boiling and thus it is most important distilled at high vacuum. The fractional distillation by using pack column to get high purity. The esters prepared by the above method is reported in table 1

Table-1

Sr. No.	Product Name	Alcohol used	Conversion	Purity checked and confirmed by G.C. [%]
1	L-Menthyl Acetate (Smell & flavor of peppermint)	L- Menthol	85 %	97.00
2	Cis-3-Hexanol Acetate (Fresh green sweet fruity apple)	Cis-3-Hexanol	90 %	99.00
3	Terpinyl acetate (Commercially important fragrance material)	Terpineol	88 %	98.00
4	Citranellol Acetate (Used in the Fragrance of lavender)	Citranellol	92 %	98.5
5	Geranieol Acetate (Fragrance material used in toiletries)	Giraneol	90 %	95.00
6	Linalool Acetate (Adulterants in essential oils)	Linalool	75 %	95.5
7	2-Phenyl Ethyl Acetate (Commercially important fragrance material)	2 Phenyl Ethanol	90 %	99.5

Conclusion-

There is a growing demand for perfumery esters, to fulfill this need the use of heterogeneous catalysts, such as Amberlyst 15 resin in the form of solid acid catalyst plays important role in the production of esters. The above process is green, clean and provides several advantages like easy separation of catalyst, high atom yield and atom economy, selectivity, and reusability.

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