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Comparative Study of Various Methods of Performing Cannizzaro Reaction and Calculating the Final Yield

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Abstract
The most often employed reaction in organic synthesis is Cannizzaro's reaction. Cannizzaro's response appears to be highly well-liked by the various universities due to its unique qualities. It can be carried out on a large or small scale and is covered in a variety of books and articles on organic synthesis.In the presence of a base, the aldehyde functional group without alpha hydrogen is transformed into carboxylic acid and alcohol. A study was carried out wherein three distinct experimental procedures were utilized to produce benzoic acid and benzoyl alcohol from the reaction between benzaldehyde and a strong base such as potassium hydroxide or sodium hydroxide. This report presents a comparative analysis of the total yield produced through three distinct experimental procedures. Mechanical stirring is the first technique, the second involves a hot water bath, and the third involves grinding. These techniques were also tested with two distinct concentrations of the same .The yields of products using two bases and three distinct processes were tallied and compared. When alcoholic sodium hydroxide was utilized as the base in the grinding process, the yield of the products increased; however, in the other two techniques that also used alcoholic sodium hydroxide, the yield of benzoic acid and benzyl alcohol was significantly lower.TLC and IR data are used to characterize every product that is obtained.

Keywords: Benzoic acid, benzyl alcohol, base, benzaldehyde, nucleophilic substitution.

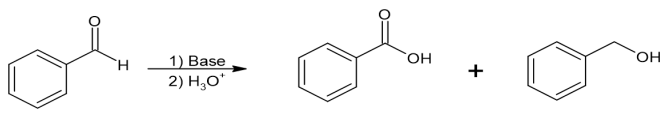
Introduction

Benzoic acid and Benzyl alcohol is an organic product formed when there is a reaction between Benzaldehyde and a Strong base like sodium hydroxide and potassium hydroxide. This reaction is called the Cannizzaro's reaction. The Cannizzaro reaction is a reaction that occurs in the compounds containing aldehyde group. The main criteria of the reaction are the absence of the alpha hydrogen. Aldehydes with alpha hydrogen are very easily prone to enolization and then aldol condensation. If there are no alpha hydrogen atoms, there can be no enolate formation and thus no aldol. The only possibility is nucleophilic attack on the aldehyde carbonyl group. When the aldehyde is made to react with the base like sodium hydroxide or potassium hydroxide there is an alcohol and a carboxylic acid formed as the product.

Benzoic acid and Benzyl alcohol have been synthesised from Benzaldehyde by three different methods. Among the three methods performed mechanical stirring method is the widely used method for the performance of Cannizzaro's reaction. The other two methods are rarely used in the performance of the reaction. The main aim of the experiment was to calculate the percentage of the final yield obtained during all the three methods and to compare them.

In continuation of our interest, the current work represents the comparative study of the final yield obtained during the three methods of performing the Cannizzaro's reaction.

Reaction Scheme



Benz aldehyde **Benzoic acid** **Benzyl alcohol**

Materials and Methods

The AR grade chemicals used to perform the reaction are benzaldehyde, sodium hydroxide, potassium hydroxide, ethyl acetate for separation of the products, sodium chloride for clear separation, and conc. Hydrochloric acid for extraction of product.

The Different Methods Employed for the Comparative Study of Cannizzaro's Reaction are Discussed Below

1. Mechanical Stirring Method

This is the widely used method for the preparation of benzoic acidandbenzyl alcohol from benzaldehyde. The mechanical stirring method includes the stirring of the reactants for a specific amount of time. The reactants include benzaldehyde and base.

A) In the first case, alcoholic potassium hydroxide is used as a base of approximately 0.04 moles (prepared by dissolving 2.2g of potassium hydroxide pellets in ethanol). The prepared alcoholic potassium hydroxide and 2ml of benzaldehyde is stirred continuously for one hour till the liquid products turn into a fine paste. The obtained paste is then taken in a separating funnel and ethyl acetate is added to which few drops of sodium chloride solution is added for the clear separation of organic and aqueous layer. To the obtained aqueous layer cconcentratedHydrochloric acid is added till the pH of the solution turns acidic. The Precipitate thus formed is then filtered using a filter paper and then dried. The dried precipitate is then recrystallized using alcohol to obtain a fine needle like crystals of benzoic acid. To the organic layer, a pinch of anhydrous sodium sulphate crystalsare added to remove the moisture content in the organic layer, filtered and heated in a water bath to evaporate ethyl acetate, after few minutes the oily layer of benzyl alcohol is obtained.

The melting point of products is determined to confirm the product formation, TLC and IR Spectrum is recorded for confirmation of product formation.

B) In the second case, alcoholic sodium hydroxide is used as base instead of potassium hydroxide for the above procedure, to compare the yield of the products in different bases used.


Mechanical Stirring Method Separation



Benzoic Acid Benzyl Alcohol TLC

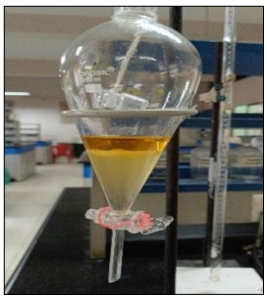
2. Heating on Water Bath

The same reaction is carried out in a water bath. The water bath heating method includes the heating of the reactants for the specific amount of time.

A) The reactants include Benzaldehyde and alcoholic potassium hydroxide. Alcoholic potassium hydroxide is prepared by dissolving 2.2g of potassium hydroxide pellets in some amount of ethanol. Then to the alcoholic potassium hydroxide and 2ml of Benzaldehyde is mixed together and kept in a water bath and stirred periodically. After 8-10 minutes the mixture becomes a fine solid paste like substance. The fine solid paste of Benzaldehyde and alcoholic potassium hydroxide is then transferred into a separating funnel. The separating funnel with the mixture is added with 20ml of ethyl acetate and sodium chloride for the clear separation of aqueous and organic layer. The separating funnel is left idle for 5-10 minutes and then aqueous and organic layer are separated put into different beakers. The beaker containing the aqueous layer is added with concentrated Hydrochloric acid till the pH of the solution turns acidic thus the precipitate formed by adding the concentrated Hydrochloric acid is the crude benzoic acid.

The crude benzoic acid precipitate is filtered out using the filter paper and then allowed to dry. The dried crude crystals are then recrystallized by dissolving them with small amount of ethanol and heating them in a water bath to remove the impurities. The solution is then filtered into a watch glass and allowed to cool. After cooling the needle like structures formed is the benzoic acid. The other beaker containing the organic layer is treated with sodium sulphate crystals to remove the excess moisture content. Then the solution is filtered into another beaker the solution is then heated to 6-8 minutes to remove the ethyl acetate content. After all the ethyl acetate is evaporated the oily layer left out in the beaker is the Benzyl alcohol. The melting points of the obtained yields are determined. The obtained yield is verified using the TLC and IR Spectrum.

B) In the second case, alcoholic sodium hydroxide is used as base instead of potassium hydroxide for the above procedure, to compare the yield of the products in different bases used.

Heating on Water Bath Separation



Benzoic Acid Benzyl Alcohol TLC

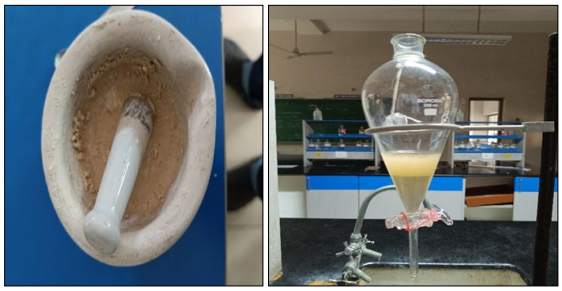
3. By Grinding Method

Grinding method is the method of preparing the benzoic acid and benzyl alcohol in a unique way. The reagents are ground in a mortar using a pestle. The method involves grinding the reagents for about 45-60 minutes.

A) The reagents include benzaldehyde and potassium hydroxide. About 1.5 grams of potassium hydroxide was taken in the mortar and about 2ml of benzaldehyde was added into it, the mixture was finely ground for about 1 hour. The solid paste that was obtained was then transferred to a separating funnel and then it is added with ethyl acetate and then with sodium chloride solution for the clear separation then the separating funnel is left idle for about 5-10 minutes then the aqueous layer and organic layer is separated into two separate beakers. The beaker containing the aqueous layer is added with concentrated hydrochloric acid till the pH of the solution turns acidic thus the precipitate formed by adding the concentrated hydrochloric acid is the crude benzoic acid.

The crude benzoic acid precipitate is filtered out using the filter paper and then allowed to dry. The dried crude crystals are then recrystallized by dissolving them with small amount of ethanol and heating them in a water bath to remove the impurities. The solution is then filtered into a watch glass and allowed to cool. After cooling the needle like structures formed is the benzoic acid. The other beaker containing the organic layer is treated with sodium sulphate crystals to remove the excess moisture content. Then the solution is filtered into another beaker the solution is then heated to 6-8 minutes to remove the ethyl acetate content. After all the ethyl acetate is evaporated the oily layer left out in the beaker is the Benzyl alcohol. The melting point of the obtained yield is determined. The products are verified using the TLC and IR spectrum.

B) In the second case, alcoholic sodium hydroxide is used as base instead of potassium hydroxide for the above procedure, to compare the yield of the products in different bases used.



Grinding Method Separation

Benzoic Acid Benzyl Alcohol TLC

IR Spectrum of the Products

A) Benzoic ACID

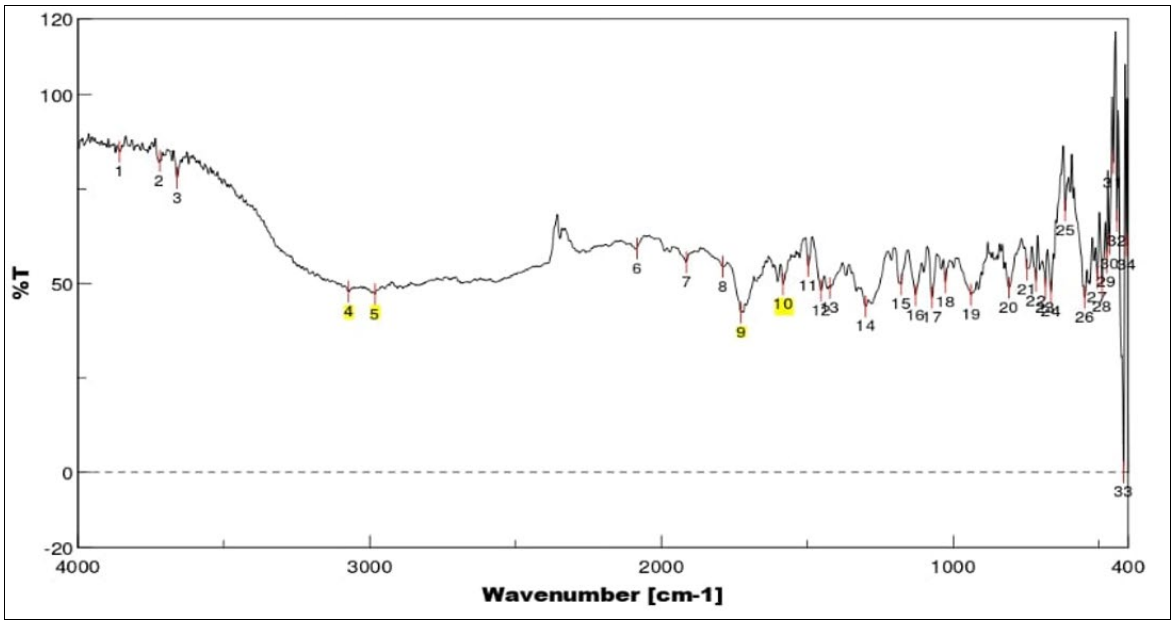


Fig 1: IR Spectrum of Benzoic Acid

B) Benzyl Alcohol

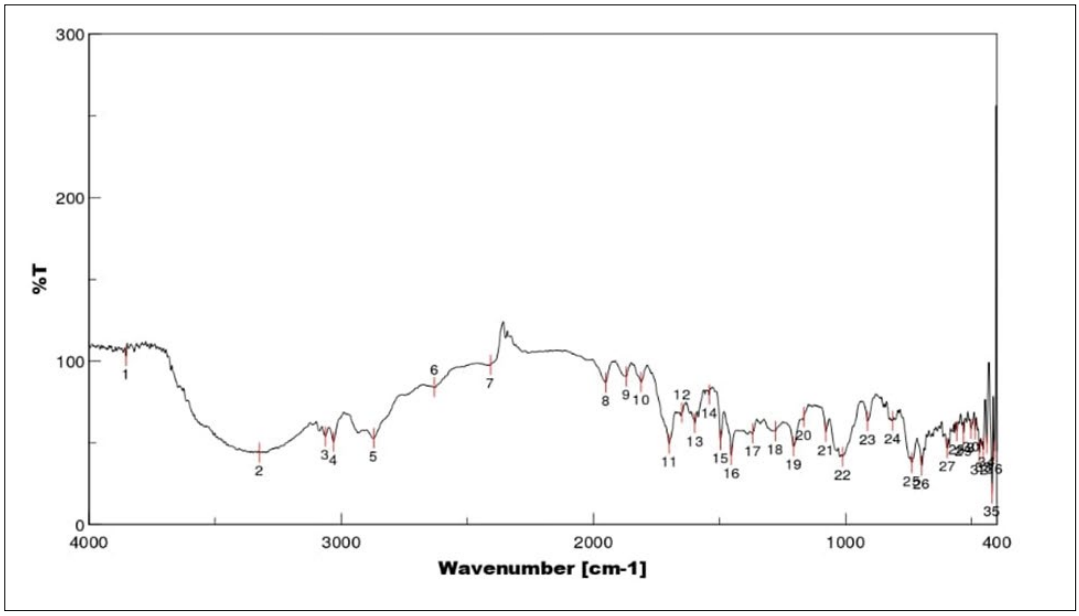


Fig 2: IR Spectrum of Benzyl Alcohol

Results and Discussion

Table 1: Comparison of Practical and Percentage Yield of Products Obtained from Three Different Method

Method	Practical Yield of Benzoic Acid	Practical Yield of Benzyl Alcohol	Percentage Yield of Benzoic Acid	Percentage Yield of Benzyl Alcohol
Mechanical Stirring Method	A) 0.65g B) 0.43g	A) 0.62mL B) 0.45mL	A) 75.58% B) 50.00%	A) 61.38% B) 44.55%
Heating on a water bath	A) 0.56g B) 0.32g	A) 0.56mL B) 0.43mL	A) 65.11% B) 37.20%	A) 55.44% B) 42.57%
Grinding method	A)0.48g B) 0.22g	A) 0.56mL B) 0.76mL	A) 55.81% B) 26.58%	A) 55.44% B) 75.24%

The above mentioned three methods are the commonly used convenient methods to prepare the Benzoic acid and benzyl alcohol from benzaldehyde. The yield obtained by these three methods is compared to the theoretical yield obtained by calculations using their molecular weights. The practical yield and percentage yield of both the products are tabulated as below,

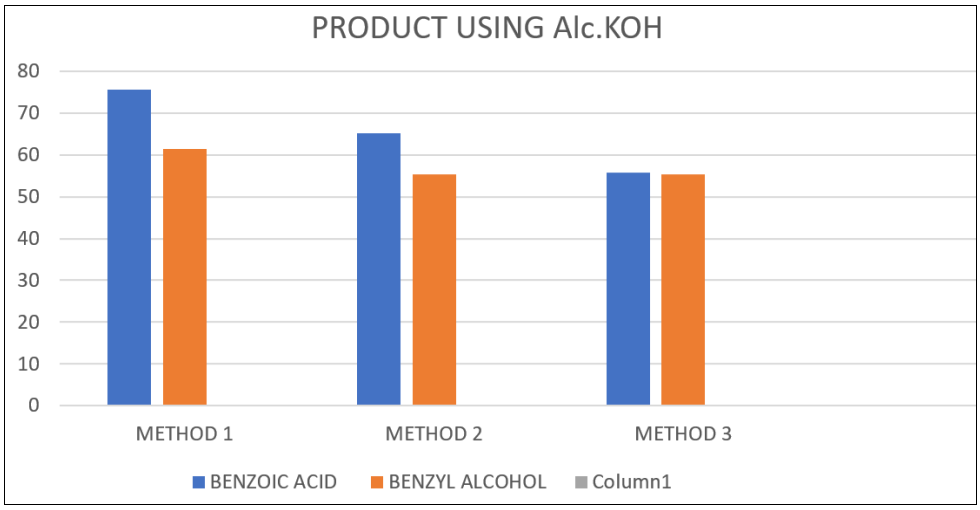


Fig 3: Representation of product yield in three methods using KOH base

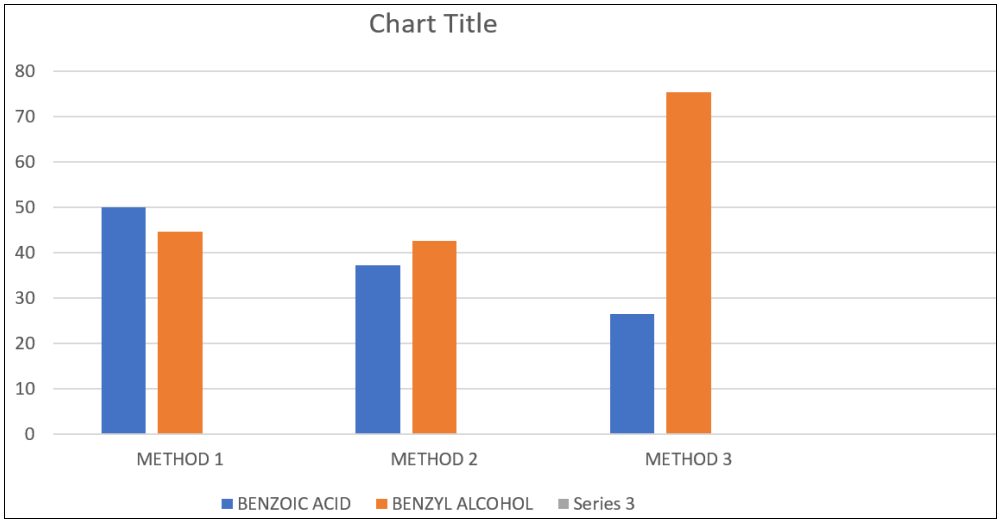


Fig 4: Representation of product yield in three methods using NaOH base

Conclusion

The comparative study of yield of benzoic acid and benzyl alcohol obtained from benzaldehyde through Cannizaro's reaction using three different methods and two different bases gives a positive result. Among the methods followed, the first method, that is mechanical stirring method gives a good yield of both benzoic acid and benzyl alcohol when alcoholic potassium hydroxide is used as a base. The yield of both the products were more when alcoholic sodium hydroxide was used as base under grinding method, but in other two methods using alcoholic sodium hydroxide the yield of both benzoic acid and benzyl alcohol was considerably less. From the above data obtained it can be concluded that the mechanical stirring method is the most convenient method for obtaining high yield of the products and among bases used alcoholic potassium hydroxide gives good yield when compared to the use of sodium hydroxide as a base.

References

1. Heathcock CH, Buse CT, Kleschick WA, Pirrung MC, Sohn JE, Lampe JJ. Org. Chem. 1980, 45(6):1066-1081.
2. Swain CG, Powell AL, Sheppard WA, Morgan CS. Cannizzaro Justus Liebigs Ann. Chem. 1853; 88:129-130.
3. Am RJ. Chem. Soc. A Noveln Type of Cannizzaro ReactionEdward M. Fry, E. Justin Wilson Jr., and C. S. Hudson. 1979; 101(13):3576-3583.
4. Geissman Org YA. React. 1944; 2:94-113.
5. List K, Limpricht H. Ann. 1854; 90:190-210.
6. Phonchaiya S, Panijpan B, Rajviroongit S, Wright T, Blanchfield JT. Chem *J.* Educ. 2009; 86:85-86.
7. Canipelle M, Landy D, Fourmentin S. *J.* Incl. Phenom Macrocycl. Chem. 2011. 69:349-353.
8. Cannizzaro S. Justus Liebigs Ann. Chem. 1853; 88:129-130.
9. Sir Richard Norman JM. Coxon. Principles of Organic Chemistry Third Edition, 1993, 654.
10. Paula Yarkins Bruce. Organic chemistry Seventh edition, 2014, 384.
11. Jerry March. Advanced Organic Chemistry: Reactions, Mechanisms, and Structure.
12. TA Geissman - Organic reactions - chemistry-chemists.com, 1944.