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Research Article

A REPORT ON PARTIAL OXIDATION OF SUCROSE

PARANAP NANDI

Department of Electrical Engineering, IIE (Wes Bengal University of Technology), BF 142, sector 1,Salt Lake City, Kolkata, West Bengal 700064.

Corresponding Author: nandiparantap@gmail.com

Abstract

Sucrose on heating at 186˚C decomposes to give brown caramel. With $KClO_3$ it gives CO_2. But it has —CH_2OH groups which may be oxidized without breaking up of the molecule. This will be a controlled oxidation which will oxidize —CH_2OH to —COOH without affecting the remaining molecule. The presence of carboxylic group can be tested using $NaHCO_3$ resulting in effervescence of CO_2. In this way a sodium salt may also be prepared. Sucrose may be oxidized using $KMnO_4$ (acidic, alkaline and neutral), $K_2Cr_2O_7$ (acidic) and acidic nitrates e.g. $Ca(NO_3)_2$, at a temperature of about **<100˚C** so that he solution doesn't start boiling. But the separation of the product is difficult as the products are all soluble. Acidic $KMnO_4$ works best as it gives a clear solution on completion of the reaction. This proves reduction of MnO_4^- has occurred to give Mn^{2+} which does not occur if only $KMnO_4$ and acid are heated. In the later case the solution retains its purple color. So sucrose acts as a reducing agent. The resultant solution formed on reaction between sucrose, $KMnO_4$ and HCl was tested with some common reagents and the observations were noted.

Keywords: Sucrose, $KMnO_4$, HNO_3, HCl, Nitrate.

Introduction

Sucrose or table sugar which is mainly used as a sweetener is a carbohydrate highly soluble in water. It has three —CH_2OH groups (refer to structure given below) which may be oxidized under suitable conditions. **In a weak oxidizing media and controlled conditions —CH_2OH may be oxidized to —CHO**. But under strong conditions —COOH is obtained. There are also 4 >CHOH which may also be oxidized to =CO group. Under highly drastic conditions CO_2 is obtained. But in this case the entire molecule will be oxidized.

Nitrates are oxidizing agents. These include $Ba(NO_3)_2$, $Ca(NO_3)_2$, $NaNO_3$, $Co(NO_3)_2$ and so on. The oxidation occurs because NO_3^- can decompose in to NO_2 and O_2. But they must be treated with strong acids in order to oxidize organic compounds. The reaction between nitrates and acids results in synthesis of HNO_3 which has strong oxidizing property. This may again be accounted on the basis of formation of **nascent oxygen, [O] which readily oxidizes different chemicals**.

However as the above mentioned nitrates are all colorless, the oxidation in an acidic medium generally is not visible to the naked eye.

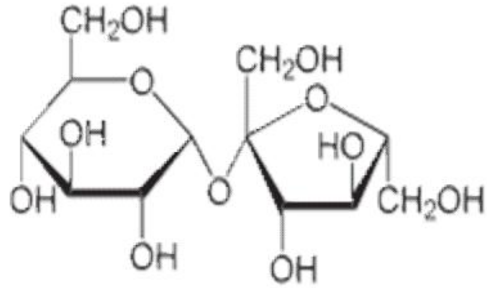
$KMnO_4$ is very good oxidizing agent in acidic, basic as well as neutral media. But the strongest conditions are created in a hot acidic media. During this reaction +7 oxidation state of Mn changes to +2. So the purple color becomes colorless. This is strong enough to oxidize even a benzene ring. In a neutral media $KMnO_4$ is reduced to MnO_2. So oxidation state changes to +4. Here the color changes to brown (characteristic color of MnO_2). When sugar is treated with alkaline $KMnO_4$, different colors are observed due to different oxidation states of Mn.

$K_2Cr_2O_7$ is a highly strong oxidizing agent in acidic media. But during oxidation orange color of $Cr_2O_7^{2-}$ changes to green Cr^{3+} resulting in a green solution. This oxidizes alcohols directly to carboxylic acids.

In this paper the oxidation of commercial sucrose derived from sugar cane using various reagents has been studied.

**This paper only deals with the methods of oxidation and the possible products. The analysis of the products using expensive reagents (e.g. Tollen's Reagent) as well as spectroscopic analysis has not been dealt with.

Structure of sucrose



Apparatus and chemicals required

1. Sucrose ($C_{12}H_{22}O_{11}$).
2. $KMnO_4$ crystals.
3. $Ba(NO_3)_2$.
4. $Ca(NO_3)_2.4H_2O$.
5. $K_2Cr_2O_7$.
6. HCl (35% concentrated).
7. HNO_3 (70% concentrated).
8. CH_3COOH (10% concentrated).
9. $NaHSO_4.H_2O$.
10. CH_3OH.
11. Methyl orange .04% indicator solution (pH 2.9–4.6).
12. Methyl red .01% indicator solution (pH 4.3–6.3).
13. pH paper (pH 2–10.5).
14. Na_2CO_3.
15. A very fine needle (used to collect $KMnO_4$).

Experimental

The properties of the acids and oxidizing agents were studied. A dilute solution of $KMnO_4$ containing about 10 granules of $KMnO_4$ (collected using a needle) was dissolved in 50ml. water. The solution turned light purple and remained transparent. To this solution, 5 drops of 35% concentrated HCl was added. The solution was heated in a conical flask. *No color change occurred*. The solution was allowed to stand for one day and yet it retained its color. This experiment proved that simply reacting $KMnO_4$ and HCl (dilute) will not produce any visible effect. So clearly the following reaction does not occur:–

$$2KMnO_4 + 16HCl \rightarrow 2KCl + 2MnCl_2 + 5Cl_2 + 8H_2O$$

The same experiment was repeated using 70% HNO_3 once and once 10% acetic acid. Again no color change was observed.

A few crystals of $Ba(NO_3)_2$ and dilute HCl were heated. The solution remained clear. The same experiment was done on $Ca(NO_3)_2$ and it produced he same results. **These experiments proved that *dilute HCl will not liberate reddish brown NO_2 from NO_3^-*.**

About 5ml. water and some sucrose were boiled in a test tube. When it dissolved, more sucrose was added. The mixture turned pale yellow. On cooling the mixture crystallized. This proved ***hot concentrated sugar solution on cooling crystallizes***.

Experiment no.1

A pinch of $Ba(NO_3)_2$ was mixed with 50ml. water and 5 drops of 35% concentrated HCl. Sucrose was added to it and boiled. When sucrose dissolved more of it was added. The solution gradually changed its color to orange yellow. The solution became sticky but remained in liquid state. A large amount of sugar dissolved in it. No crystallization took place.

Experiment no. 2

The above experiment was repeated by using $Ca(NO_3)_2.4H_2O$ instead of barium nitrate. A large amount of sucrose dissolved but *unlike barium nitrate, no coloration was noticed*.

Experiment no. 3

A pinch of $K_2Cr_2O_7$, 5drops of 70% concentrated HNO_3 and 50ml. water were boiled with sugar. The orange color of $K_2Cr_2O_7$ turned dark brown. Green color was not noticed. No precipitate was obtained.

Experiment no. 4

A small amount of $KMnO_4$ (less than a pinch) was added to 50ml. water. Sucrose was added to it and boiled. The mixture was stirred repeatedly to ensure uniform mixing. The solution turned deep red. Slowly a brown precipitate of MnO_2 was obtained.

Experiment no. 5

A very small quantity of $KMnO_4$ collected on the edge of a fine needle was dissolved in 50ml. water. 5drops of 35% HCl were added to it. Sucrose was added to it and the solution was boiled with continuous stirring. The purple color gradually turned deep red then brown. Ultimately the solution became colorless.

Experiment no. 6

The above experiment (no. 5) was repeated using 5 drops of 70% HNO_3 instead of HCl. The same observations were made.

Experiment no. 7

A special experiment was performed using very minute quantity of $KMnO_4$ (just enough to turn 50ml. water pink) acidified with 2 drops of 35% HCl and two pinches of sucrose. The resultant solution was decolorized by heating. The colorless solution was cooled and about 3 ml. of it was collected in 5 separate test tubes. The following tests were performed on them:–

Test A
A pH paper was soaked in the first solution.
Test B
To the second sample one drop of methyl orange was added.
Test C
To the third test tube one drop of methyl red was added.
Test D
To the fourth sample 5-6 grains of anhydrous Na_2CO_3 was added and the solution was allowed to stand for a while. The resultant solution was tested with pH paper.

Observations:

When sucrose was heated with acidified solution of *barium nitrate* the solution turned yellow. The solution had both H^+ and NO_3^-. So it was oxidizing. NO_2 was not present because if it were so, it would escape on heating. The solution dissolved a very large amount of sugar. The color might have been due to partial oxidation of sucrose or breaking up of the molecule in to smaller compounds. But clearly it was not sucrose because pure sucrose would crystallize at such a high concentration.

$Ca(NO_3)_2.4H_2O$ showed somewhat similar results except for the color. It showed almost no coloration (a very faint yellow color was observed).

$K_2Cr_2O_7$ when heated with HNO_3 (without sucrose) turns reddish brown. When heated with sucrose it showed the same results.

Neutral $KMnO_4$ with any reducing agent e.g. some organic compound turns brown. This is due to the reduction of MnO_4^- (purple) to MnO_2 (brown).

In acidic media, H^+ reacted with MnO_2 to give a colorless solution of Mn^{2+}. Hence the solution got decolorized. This occurred for both HCl and HNO_3.

When he tests were performed the following observations were made:–

A. The pH paper turned pink.
B. Methyl orange turned light pink.
C. Methyl red turned light pink.
D. No effervescence was noticed on addition of Na_2CO_3. But when the resultant solution was tested with pH paper, the paper turned deep blue.

Results and discussion

The four **>CHOH** groups in sucrose must have been oxidized by NO_3^- from barium nitrate in the acidic media caused by HCl. The volume of water in the solution increased. But *acidic nitrate not being a very strong oxidizing agent can not result in synthesis of carboxylic acid*. The only possible product is a ketone i.e. **>C=O**. But all 4 **>CHOH** groups were not oxidized because if it were so the product would become insoluble. Also crystals would form. But as it was not so **it may be inferred that one of the secondary alcoholic groups was oxidized to a ketonic group**.

$Ca(NO_3)_2.4H_2O$ must have resulted in the same thing. But the colors were different owing to the **different absorption of wavelength by Ca^{2+} and Ba^{2+}**.

$K_2Cr_2O_7$ and HNO_3 both are very strong oxidizing agents and on heating they create drastic conditions. So instead of oxidation of —CH_2OH or >CHOH it oxidizes the entire molecule to CO_2 and H_2O. The reaction may be compared with burning of sucrose in chloric acid given by

$$8\ HClO_3 + C_{12}H_{22}O_{11} \rightarrow 11\ H_2O + 12\ CO_2 + 8\ HCl \quad (1)$$

A small portion of sucrose might have undergone **nitration** resulting in a reddish brown solution.

$KMnO_4$ in a neutral media changes oxidation state of Mn from +7 to +4. This is enough to oxidize —CH_2OH groups to —CHO groups. A small fraction of sucrose may be oxidized to —COOH. But this is negligible.

In acidic media +7 oxidation state of Mn changes to +2. So this solution is enough to oxidize —CH_2OH to —COOH. As Mn^{2+} is soluble and colorless the solution becomes clear. This occurs for both HCl and HNO_3. **A solution of $KMnO_4$ and HNO_3 is not drastic enough to cause nitration of sucrose**. The resultant carboxylic groups form hydrogen bonds with water and the product is water soluble.

The pH paper shows an orange color at a pH of 4 and a red color at a pH of 2. But the color was midway i.e. pink. Also the pH depends on the amount of acid added. As only 2 drops of HCl were added, the pH was midway between 2&4. Addition of more acid might result in a lower pH.

Methyl orange works between rated pH of 2.9-4.6 and changes its color from **orange red to orange yellow**. But when the resultant solution was tested the color obtained was light pink. This too indicated that **$2<pH<4$**.

Methyl red operates between pH 4.3-6.3. It changes its color from red o yellow. But in this case too the color changed o light pink. Hence it may be inferred that **$2<pH<4$**.

Normally acids show effervescence with Na_2CO_3. Though $NaHCO_3$ is used instead of Na_2CO_3, sodium carbonate also works. In this case however sodium carbonate did not show any response. This might have been due to the fact that pH was no low enough to cause any visible reaction. E.g. Phenol is also acidic but it can not be tested with Na_2CO_3. It is tested with $FeCl_3$ which shows violet coloration. But **sodium carbonate being alkaline in nature increased the pH of the solution to as high as ≈11 which turned he pH paper blue**.

So finally it may be inferred that oxidation of sucrose is possible without complete burning of the molecule (as seen in he case of chloric acid) to give aldehydes and carboxylic acids with suitable reagents like $KMnO_4$. A mineral acid like HCl or HNO_3 when added increases the efficiency of oxidation. But addition of an acid results in formation of Mn^{2+} salt which is soluble and hence is difficult to separate. The excess acid also makes the solution acidic.

Conclusion

Sucrose can be oxidized using NO_3^- (acidic), $Cr_2O_7^{2-}$ (acidic) and MnO_4^- (both acidic and neutral). But nitrates are too weak while dichromate is extremely strong. So both of them are not convenient to use. MnO_4^- can serve he purpose very well as it is neither too strong, nor too weak. Neutral $KMnO_4$ can be used effectively because the insoluble MnO_2 can be filtered out using a filter paper. **The filtered solution must be almost neutral if carboxylic acid group is absent**. The presence of carboxylic acid group may be detected using $NaHCO_3$. Reagents like $HClO_3$ or KNO_3 should not be used as they will result in combustion of sucrose. In case of acidification of $KMnO_4$ only mineral acids should be used. Organic acids like CH_3COOH, $H_2C_2O_4$ are not suitable for acidification because MnO_2 is insoluble in these acids. Hence neutral $KMnO_4$ shows best results as: —

I. The resultant solution is almost neutral.
II. Insoluble MnO_2 may be easily filtered.
III. Neutral $KMnO_4$ does not cause excessive oxidation of sucrose.

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References

The reaction labeled **(1)** and the structure of sucrose molecule have been taken from Wikipedia, the free encyclopedia.