

SIMULTANEOUS SYNTHESIS OF FORMIC ACID AND FORMALDEHYDE

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Abstract- The formaldehyde is prepared by catalytic oxidation of methanol by passing it over copper. This is the simple method to produce formaldehyde. Here, the oxidation process is very much fast that some of the produced formaldehyde is converted into formic acid. The formic acid is produced in very less amount as compared to main product but formic acid is also produced in its pure form and can be collected easily. The collected amount of formic acid was tested through some laboratory tests to check the acidic properties of the formic acid. Besides this, the formic acid is also analyzed by IR spectroscopy.

Keywords- Catalytic oxidation, Formic acid

1. INTRODUCTION

Formic acid is naturally occurred in the stings of different insects, like honey bee, ant etc. It behaves both as an acid and an aldehyde as its carboxylic group is not linked to the hydrocarbon radical. Its name is derived from the Latin word “**Formica rufa**” which means “**red ant**”, in which it exists as a free acid^[1]. Formic acid is also found in some natural products, for example, the juice of hairs of stinging nettle plant, juice of ginkgo tree, fruits, juice of ants and some of the caterpillars of the order **Hymenoptera**.^[2]

In 1671, formic acid was firstly extracted from the ants. John Ray distilled number of dead ants and got the chemical compound which is further named as Formic Acid^[3].

In laboratory, the formic acid was prepared by the French chemist **Joseph Gay-Lussac**, he used the hydrocyanic acid as starting material.

It is also prepared by combining methanol and carbon dioxide in the presence of a strong base results into methyl formate, like this;



In industry, this reaction is performed in the liquid phase at elevated pressure. Typical reaction conditions are 80°C and 40 atm. The most widely-used base is sodium methoxide. Hydrolysis of the methyl formate produces formic acid.



In 2003, Ezhova,^[5] synthesized the formic acid in the presence of Wilkinson complex and excess of PPh₃ at high temperature. Rhodium complex catalyst properties depend on reaction conditions. Catalyst deactivation was studied by kinetic method and ³¹P NMR spectroscopy, and method for its stabilization was found. In 2008, Yingpu and Gongxuan,^[6] prepared formic acid and carbon dioxide free hydrogen from formaldehyde by nano-metal catalyst at room temperature and atmospheric pressure. Among Pt, Au, Ni, and Cu nano-metal particles, nano-Cu catalyst exhibited the highest activity and the long-term stability.

2. EXPERIMENTAL WORK

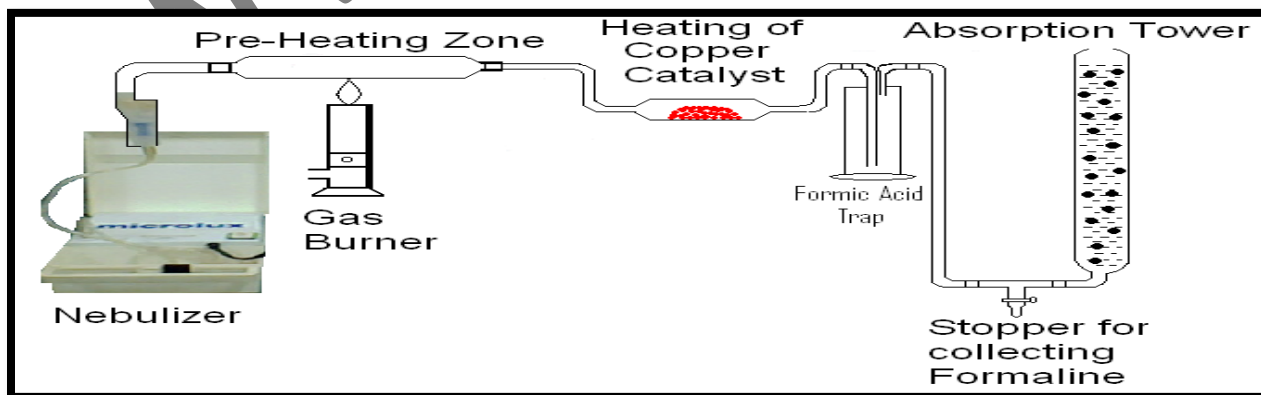


Fig 2.1 Apparatus for the Synthesis of Formaldehyde and Formic Acid

2.1 Reagents

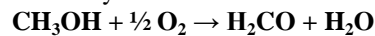
Methanol (commercial), copper, calcium chloride,

2.2 Apparatus

A schematic assembly consists of nebulizer, tube having copper, absorption tower is shown in fig. 2.1.

3. METHOD

Methanol is purified by using calcium chloride as dehydrating agent. Methanol is atomized and mixed with air by using nebulizer; the vapors are heated in pre-heating zone and then passed over the copper bed. With the help of this catalyst, methanol is converted into the formaldehyde.



Meanwhile, due to high temperature, some amount of the formaldehyde is undergoes further oxidation process by which it is converted into the formic acid before goes into the absorption tower for storage.



4. CHARACTERIZATION OF FORMIC ACID

The formic acid is stored or collected into the separate jar by placing the jar into ice bath. The collected formic acid is analyzed by performing some tests.

- The prepared formic acid acidity was tested by applying the pH paper and the color of the paper was compared with the formic acid present in the laboratory. Both having the same color indicated on pH bar.
- The test was also performed with the pH meter and the results of both acids prepared and existed were compared. There was no extinct difference between both.
- Lastly, most differentiated analysis was performed by taking IR spectra of prepared and existed formic acid. The IR results present the clear picture of the product formed.
- These spectrum results are shown here,

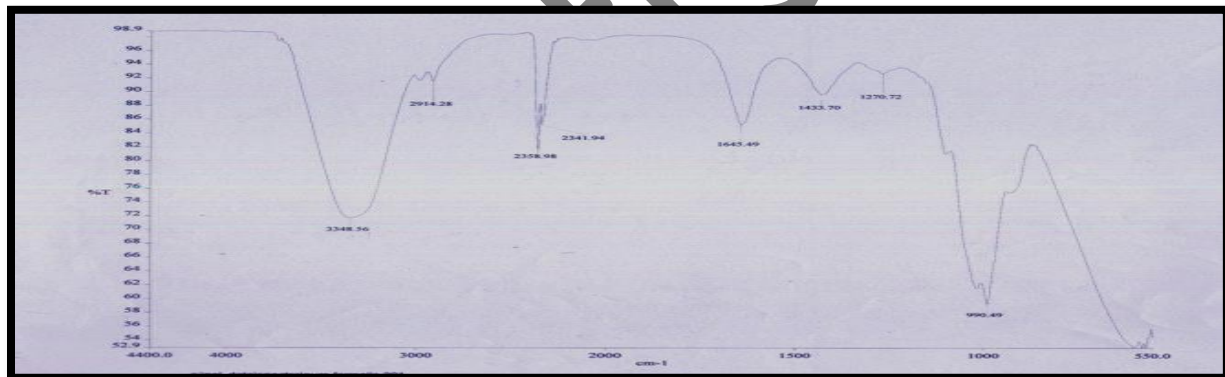


Fig. 4.1 IR spectra of Pure Formic Acid

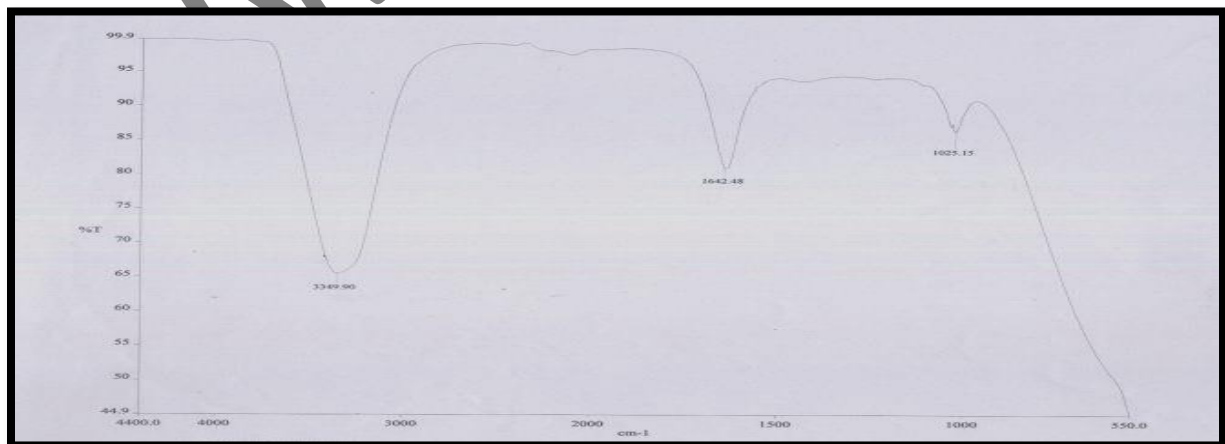


Fig. 4.2 IR Spectra of Prepared Formic Acid

5. RESULTS AND DISCUSSION

The Boiling Point of pure formic acid was 101°C, while of obtained formic acid was 100°C.

The obtained product (Formic Acid) was compared with the pure formic acid by infra-red spectrum as shown above, the values are:

- The Carbonyl Peak of pure Formic acid was shown at 1695.18 cm^{-1} . The Carbonyl peak of obtained formic acid was shown at 1642.77 cm^{-1} .
- The obtained formic acid was shown the peak at 3342.34 cm^{-1} due to the impurities of water evolved during condensation.

6. FINDINGS

The formic acid which was obtained during the process was pure as the formic acid was available in laboratories. Its concentration was checked by taking infra-red spectrum and boiling points with small differences. The pH of formic acid was same as that of pure formic acid.

Although, it is produced in small amount but its quantity could be increased by increased the length and catalyst column and catalyst concentration. This formic acid was produced along with the formaldehyde; hence, it was separated in another bottle to produce the formaldehyde free from formic acid.

About, 3ml of formic acid was prepared from the 10 ml of methanol added in the nebulizer. The formic acid readily converted by the oxidation of formaldehyde.

CONCLUSION

- The formic acid was produced in less quantity but contained purity same as the pure formic acid.
- The purity of the catalyst was also of great importance. It was observed that the catalytic oxidation with blackish layer of copper oxide lowered the rate of methanol oxidation.
- The yield of formic acid can be increased by increasing the length of the catalyst column.

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