EVIDENCE OF FORMATION OF HYDROGEN PEROXIDE BY RADICAL REACTION INDUCED BY MICROWAVE IRRADIATION OF SODIUM HYDROXIDE

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أمكن الحصول على فوق أكسيد الهيدروجين (H_2O_2) بتشعيع هيدروكسيد الصوديوم بموجات المايكروويف تحت ضغط منخفض مع إمرار تيار من غاز النتروجين؛ وتم التأكد من ذلك بتمريره إلى محلول مائي حمضي من يوديد البوتاسيوم في وجود دليل النشا.

Microwave irradiation of sodium hydroxide (NaOH) under vacuum and a stream of nitrogen gas produced catalytic quantities of hydrogen peroxide (H_2O_2) that was confirmed by passing it into acidified potassium iodide (KI) aqueous solution with starch indicator.

Key Words: Microwave Irradiation, Sodium Hydroxide, Iodometric Method.

INTRODUCTION

Hydrogen Peroxide is manufactured today almost exclusively by the autoxidation of 2-ethyl-9,10-dihydroxy anthracene to 2-ethylanthraquinone and hydrogen peroxide [1]. Several methods of producing hydrogen peroxide in catalytic quantities have been reported. It was found that leaving water in the sun light for four hours generates hydrogen peroxide in ppb quantities [2]. Cation of aqueous media in the presence of boron- doped diamond electrode allows the electrochemical reduction of oxygen molecule to hydrogen peroxide [3]. UV irradiation of fluorinated TiO_2 suspensions in water in the presence of oxygen and formic acid or phenol as a hole scave leeds to the production of hydrogen peroxide in ppm quantities [4].

The use of domestic microwave oven as a source of energy for enhancing chemical reaction has been used for the past decade and still growing [5-7]. Several advantages of microwave (MW) irradiation of polar compounds include shorter time, high yield and attain reaction hard to be achieved with conventional heating [8].

Several methods were used to detect the production of hydrogen peroxide depending on the conditions of the reaction (2,4) but the iodometric method [9]was suitable for our work because of its stability and clear color change which proves the production of hydrogen peroxide.

In this present work, we report an evidence of formation of hydrogen peroxide upon irradiation of sodium hydroxide pellets inside a domestic microwave oven under pressure and nitrogen gas current. The iodide solution showed a clear colour change to dark pink.

EXPERIMENTAL

Instruments:

Generation of hydrogen peroxide was conducted in solid phase using modified domestic house hold microwave oven (Daewoo model KOR-861H,1200 W) and irradiation was done at 1200W (100%). Two holes 5 cm apart were opened in the upper back of the oven as shown in (Fig. 1) that will fit a U tube of 10 cm length.

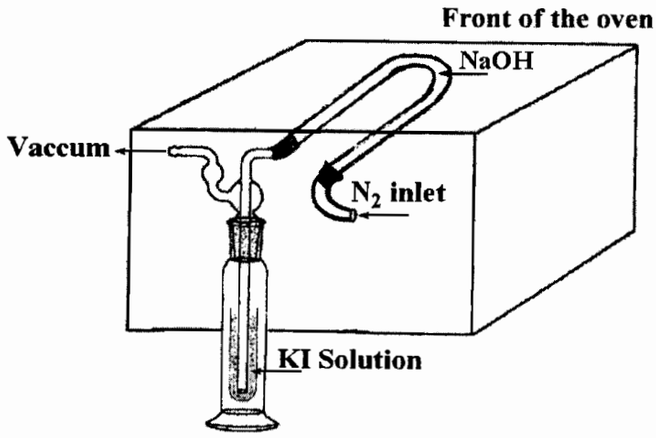

Fig. 1: Setup of the experiment.

Chemicals:

All chemicals used in this work were of AR grade and without any further purification. sodium hydroxide (NaOH) (Winlab,UK 98.0%), sulphuric acid(H_2SO_4) (BDH,England,Analar), potassium iodide (BDH,England,98.0%), nitrogen gas of high purity(SIGC, Saudi Arabia), distiled water and starch indicator freshly prepared in the lab as 5% starch solution.

Procedure:

A 5 % acidified aqueous KI stock solution was prepared by dissolving 4g of KI in 95cm^3 distilled water plus 5cm^3 0.5 M H_2SO_4. Four samples of the solution were made by placing 5cm^3 of the stock solution in a test tube and a few drops of starch solution were added in each. One test tube was covered with aluminum foil to role out light effect and left without any further treatment as a reference.

To one of the other three, a drop of 30 % H_2O_2 solution was added to see the change of colour and check the validity of potassium iodide (KI) solution. The second was placed in ultrasound cleaning bath to check the colour change at low concentration of Hydrogen peroxide. The third was placed in a purging tube and covered with aluminium foil and purge of three times with nitrogen gas. While keeping the vacuum, light current of nitrogen was passed to enhance the transfer of product and of the U tube until the light yellow solution colour changes to dark pink or black. Cracking and /or melting of the glass tubes was experienced repeatedly due to high temperature generation.

RESULTS AND DISCUSSION

It is well known that iodide in acid solution reacts with hydrogen peroxide to form iodine according to the following equation [10].

$$H_2O_2 + 2I^- + 2H^+ \longrightarrow I_2 + 2H_2O$$

The liberation of I_2 is the cause of colour change. We believe that the irradiation under the specified conditions leeds to the formation of hydrogen peroxide through the following mechanism:

$$NaOH \longrightarrow Na^\bullet + {}^\bullet OH$$

$$2{}^\bullet OH \longrightarrow H_2O_2$$

$$H_2O_2 \longrightarrow H_2O + {}^\bullet O$$

$$2Na^\bullet + {}^\bullet O \longrightarrow Na_2O$$

The fraction of hydrogen peroxide in the second step or the oxygen radical originated from the decomposition of hydrogen peroxide in the third step is the cause of the iodide oxidation. Running a stream of nitrogen gas during

continuous evacuation decreases the residence time of hydrogen peroxide in the U tube which might decrease the probability of the formation of oxygen radical via the third step. The addition of water vapor did not regenerate hydrogen peroxide in a cyclic process that means the source of hydrogen peroxide is the decomposition of sodium hydroxide(NaOH) caused by the irradiation.

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REFERENCES

[1] C.A. Crampton, G. Faber, J.P. Leaver and S. Schell, The manufacture, The Modern Inorganic Chemicals Industry, pages. 232-66. Chemical Society Special Publications (RSC), No. 31(1977).

[2] Mohammad A. Abdalla and Hassan M. Al-Swaidan, Analytical Letters, **22** (7), 1729-1742 (1989).

[3] Richard G. Compton, Frank Marken, Christian H. Goeting, Ross A.J. Mckeown, John S. Foord, G. Scar Sbrook, R.S. Sussmann and F.J. White Head, Chem.Commun, 1961 (1998).

[4] Valter Maurino, Claudio Mineeo, Giuseppe Moriella and Ezio Pelizz Etti, Chem. Commun., 2627-2629(2005).

[5] S. Caddick, Tetrahedron, 10403 (1995).

[6] R.S. Verma, Green Chemistry, **43,** 1 (1999).

[7] C.O. Kappe, Angew Chem. Int. Ed., **43,** 6250 (2004).

[8] Pelle Lidstrom, Jacson Tierney, Bernard Wathey and Jacob Westman, Tetrahedron, **57**, 9225-9283 (2001).

[9] Timothy J. Mason, Sonochemistry, page 36, Oxford University press Oxford (1999).