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EXTRACTION AND CHARACETRIZATION OF FURFURAL FROM WASTE OMANI DATE SEEDS

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Abstract

Purpose: Furfural ($C_5H_4O_2$), is an economic and business product in European countries such as America and Australia. Previous years have seen a remarkable increase in the number of palm trees in the Arabian region. The percentage of furfural present in dates seed is around 30%. This paper outline the extraction of furfural from waste Omani date seeds.

Methodology: Date seeds were washed, sun dried, heated at 100^0C, grinded, powdered, and mixed with solvent n-hexane for one day. Then filtered and filtrate was subjected to simple distillation at 60^0C in round bottom flask. Furfural was recovered in round bottom flask and solvent was recovered in other beaker. This hexane was reused for furfural extraction from other batches.

Findings: Extracted product was characterized by Carbon NMR, and Proton NMS. The Carbon NMR result the experiments were carried out in Bruker Avance III HD 700 MHz spectrometer equipped with 5mm TCI H/C/N cryoprobe. The proton NMR experiment was run using zg30 pulse program operating at 700.13 MHz. Occurrence of C-NMR peaks at 127, 131, 173 ppm confirms the presence of carbon atoms in furfural ring. And presence of H-NMR peaks between 4 to 8 ppm confirms the presence of furfural protons.

Social Implications: Furfural substance is used in a number of the important chemical industries such as nylon, plastic, ratings that protect the metals from corrosion, solvents, adhesive, medicines, and plastics and is used in the industry of insecticides, fungicides, anti-microbe, and antiseptics. Therefore, it is widely used in the petrol refinery laboratories to treat the bad Carbon and different Sulfuric combinations existing in the lube oils and it is used in the operations of refining some types of fuel as well, such as diesel.

Originality/ Novelty: This study is done on Omani date seeds at Caledonian College of Engineering in Chemical Analysis Lab. Extraction product was characterized in Central Analytical and Applied Research Unit at Sultan Qaboos University, Muscat.

Keywords: Furfural, Furan, 2- Carbaldehyde Furan, 3- Carbaldehyde, date seed.

INTRODUCTION

Furfural is an aldehyde, organic, oily and liquid substance. It is a colorless, oily liquid with the odor of almonds, changes to the dark yellow or amber color when exposed to air. Its systematic name Furan-2-carbaldehyde and sometimes it is called furfuraldehyde. Its chemical formula is $C_5H_4O_2$, (See figure 1) with molecular mass of 96.09 gm/mol, density 1.16 gm/cm^2, melting point is -36.5^0C, boiling point is 161.7^0C. Its smell resembles the bean oil smell or the smell of Benzene Aldehyde. **(Adams and Voorhees, 2011) (Kamalu and Ogbome, 2008)**

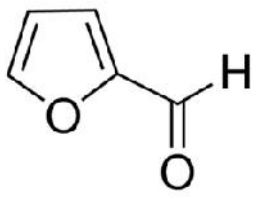

Figure 1. Chemical structure of Furfural

(**Dowson and Aten, 1962**) has made an accurate chemical analysis for the date seeds which showed its richness of some very important fat acids. The study was later supported by the analysis by (**Sawaya, 1986**).

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Table 1. Fatty Acid percentage in the date seeds

Type of Fatty Acids	Percentage
Cbric acid	0.7%
Carrinic acid	0.5%
Lournic acid	24.2%
Mirestic acid	9.3%
Balmatic acid	9.2%
Ualic/ilmic acid	25.2%
Citric acid	03.02%

Various parts of date seeds have different percentage of furfural. The following table 2 shows the percentage of Furfural that can be extracted from the different parts of the Palm tree, estimated on the basis of the hard substance.

Table 2. Percentage of Furfural extracted from the different parts of the Palm tree

Palm Tree Part	Furfural percentage
Dates' seeds	30.00%
stem	16.70%
Palm	16.40%
Hmarich	14.50%
Palm tree fiber	12.70%
Palm tree trunk	11.70%
Palm leaves	08.50%

The world market for furfural is around 300,000 tones / year with annual growth of about 2 % per year. Furfural is used in a number of the important chemical industries such as nylon, plastic, ratings that protect the metals from corrosion, solvents, adhesive, medicines, insecticides, fungicides, anti-microbe and antiseptics. Also, it is considered as one of the selective solvents. Therefore, it is used widely in the petrol refinery laboratories to treat the bad carbon and different sulfuric combinations existing in the lube oils. It is also used in the operations of refining some types of fuel as well, such as diesel. (**Dowson and Aten, 1962**) (**Sawaya, 1986**).

The industrial application furfural had seen a big increase during the few past decades, where it used in the manufacture of paints, sticking materials, perfumes, textiles, paper, anti-rust, human and veterinary medicines. Also, the Furfural is considered an important preliminary substance to manufacture a big number of important chemical combinations of multi and vital industrial usages as it is used to produce the furfural alcohol. The rubber factories use it to produce the hydro furan and Butadiene required for the manufacture of the industrial rubber. Also, the lab experiments showed that treating the rubber with the furfural accelerates the process where it results in producing hard rubber that bears the hard operational and environmental conditions.

Arabs have been using the date's seeds in a great number of various life purposes. Also, the have extracted medical oils that were used to treat some diseases such as Gout, joints pain and rheumatoid, and in preparation of a special drink similar to coffee; by roasting the seeds then boiled them in water. This drink was thought that it has important remedial characteristics.

From another side, the dates' seeds have been used as an animal food by mixing it with barley and sesame cake and corn. The studies have showed the importance extent of this mix, as the weight rates of the fattening animals were increased because the seeds contain the growth hormones which very clearly participate in the animal's growth.

Date seeds contain important nutritious components for the human body such as carbohydrates, fats, proteins and minerals. Seeds are cooked after roasting and then consumed as human food beneficial to the body. Later it has been discovered that it contains

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considerable quantities of fibers which protect the human body from indigestion and constipation. The following table 3 shows the nutritional composition of date seeds.

Table 3. Nutritional composition of the dates' seeds

Substance	Percentage
Carbohydrates	62.51%
Fibers	16.20%
Fats	08.49%
Humidity	06.46%
Salts	00.50%
Protein	00.22%
Others	05.62%

And a big number of the world countries are interested in producing the Furfural of which the price of one ton in 2008 was about $1,700 **(Dalin Trading, 2004).** The previous years have seen a remarkable increase in the number of palm trees in the Arabian region including United Arab Emirate, Oman and other Gulf countries. The date seeds which are 10 to 15% of the whole date's weight are considered one of the most important surpluses resulting from dates and which represent an important economic value and an environmental issue at the same time if accumulated in vast numbers in the nature. Hence, the human being has utilized the date seeds in a big number of important industries and products of economic feasibility which open wide horizons for promising industries that depend on date seeds and dates surplus in Arabian territory. (**Dowson and Aten, 1962**) (**Sawaya, 1986**).

AREA FOR STUDY

Caledonian College of Engineering in chemical analysis lab with help of Sultan Qaboos University in Central Analytical and Applied Research Unit, for analyzing the product sample.

METHODOLOGY

Date seeds were collected from local market in Muscat. Seeds were washed several times with distilled water to remove soluble, insoluble parts, dust etc and then sun dried for one day. Dry seeds were heated in electric oven at 100^0C to remove moisture from the seeds. These seeds were grinded and powered using mill machine so that it can be mixed well with solvent. Powder was kept in n-hexane solvent for one full day at room temperature with occasional shaking. Liquid portion was filtered off [Figure 2(a)]. And the process was repeated for two more times so that maximum furfural dissolves on solvent. The liquid portion is now transferred to round bottom flask and subjected to simple distillation at 60^0C as per setup shown in figure 2 (b). Process was carried at this temperature because boiling point of n-hexane is 68^0C. Furfural product was recovered from the round bottom flask. n-hexane was recovered and reused for next batch of extraction. The product sample was characterized by CMR and NMR at Central Analytical and Applied Research Unit in Sultan Qaboos University, Oman.

Figure 2 (a): Filter the date seed from solvent n-hexane

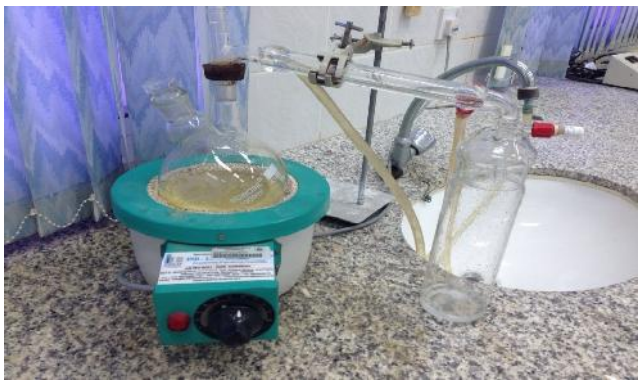

Figure 2 (b): Simple Distillation

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RESULT AND DISCUSSION

Carbon NMR testing was carried out in Bruker Avance III HD 700 MHz spectrometer equipped with 5mm TCI H/C/N cryoprobe. The proton NMR experiment was run using zg30 pulse program operating at 700.13 MHz Acquisition parameter was as follows: 90° proton pulse width of 8.00 µs, a relaxation delay of 1 s, 128 scans. The Spectrum was recorded in CDCl3 at 298K and processed using TOPSPIN 3.2 software.

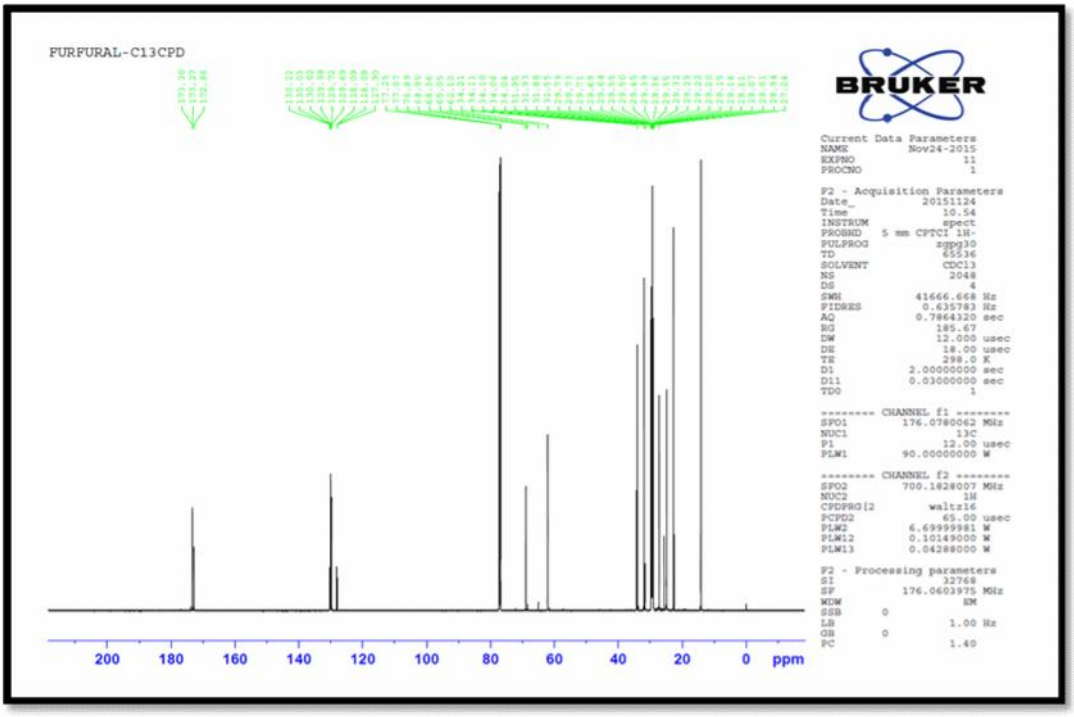

Figure 3. Carbon NMR of the Product

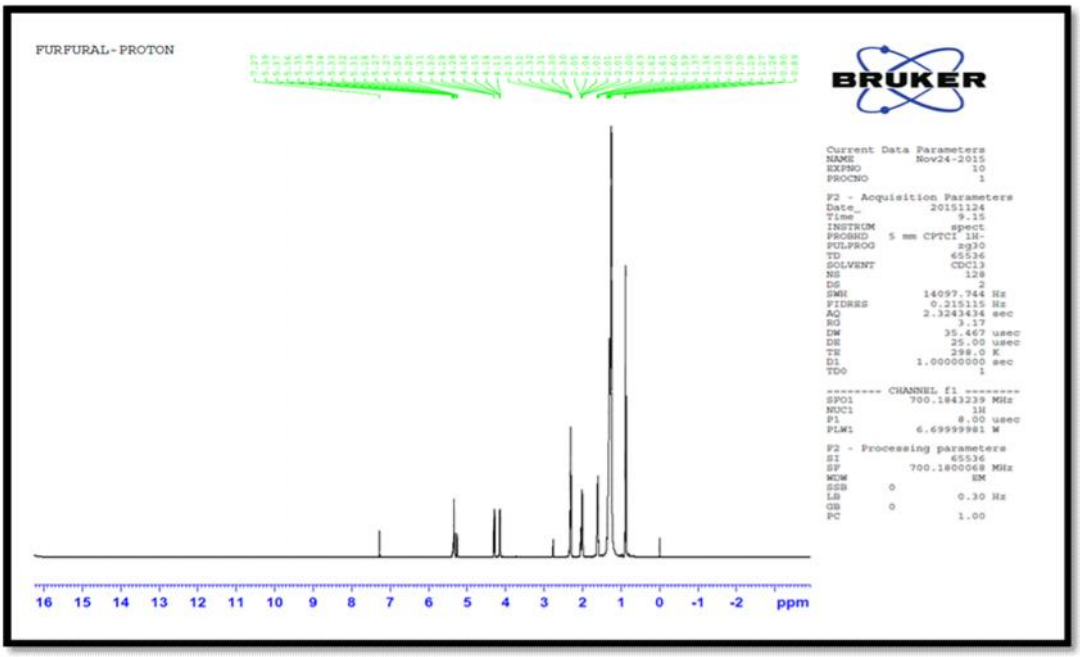

Figure 4. Proton NMR of the Product

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In above figure 4, Carbon NMR of the furfural is shown. Presence of peaks at 127, 131, 173 ppm is showing presence of carbon atoms in furfural ring. For NMR Proton result experiments were carried out in Bruker Avance III HD 700 MHz spectrometer equipped with 5mm TCI H/C/N cryoprobe. As clear from figure 5. The presence of furfural protons is confirmed by the presence of peaks in the range of 4 to 8 ppm.

CONCLUSIONS AND RECOMMENDATION

In current project furfural was successfully extracted from the waste Omani date seeds. C-NMR and H-NMR results confirm the presence of furfural in the extraction product. Solvent n-hexane was recovered during the project and reused. In Oman about 50% of the total agricultural area and 83% of the total area of fruit trees is covered by dates. So it is economical to start industry based on furfural extraction. In this study, percentage purity of product was not measured and few more confirmatory tests like IR and GC-MS could also be conducted. In next study authors are proposing to present the plant design for furfural extraction using waste Omani date seeds.

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