Experimental Study to Improve Bio-Ethanol Yield from Iraq Dates Crop

دراسة عملية لتحسين إنتاج الايثانول الحيوي من التمور العراقية

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ABSTRACT:

Bio-ethanol can be produced from different agricultural crops such as sugar cane, beat root, dates, etc. In Iraq the Zahdi species of dates is produced in very large quantities with good sugar content so it is chosen for the production of bio-ethanol. This research presents the possibility of improving the bio-ethanol yield from dates and to reduce the production cost. A special rig is designed and assembled for this purpose. Dates juice is available in local markets so it is used as the raw material for the production. Ordinary production process includes many stages namely; fermentation, distillation and dehydration. All these stages are carried out under atmospheric pressure.

Two techniques are tested in this research. The first technique is to add another stage for the production stages which is a pre-treatment heating process of date juice in a microwave oven before the fermentation process in order to reduce the fermentation time. The second technique is to carry out the distillation and dehydration stages under vacuum pressure. Two vacuum pressures are tested namely 0.5 and 0.7 bar vacuum.

It is found that the pre-treatment of the juice in the oven reduces the fermentation time. Three residence times in the oven are tested namely (10, 20 and 30 min) at 140 ^{o}C. When the residence time exceeds 30 min the sample burns in the oven. The fermentation time for these residence times decreases by 8.3, 20.8 and 29%

respectively. The reduction in fermentation time reduces power consumption and hence production cost.

The fermentation process is followed by the distillation process under vacuum pressure, which includes three stages. Two experiments are done one under a vacuum pressure of 0.7 bar while the other under a vacuum pressure of 0.5 bar. Different evaporation temperatures are tested for each stage to obtain the best temperature for each stage which gives the shortest time and the highest concentration. It is found that carrying out the distillation process under vacuum reduces the total distillation time by about 32%. As the vacuum pressure increases the total distillation time decreases due to lower evaporation temperature. Finally, dehydration process under vacuum pressure is the most important and difficult process because it takes a longer time to produce the required concentration of ethanol. It is found that carrying out the dehydration process under vacuum reduces the total dehydration time about 33%. This process involves the use of different types of drying materials (ethylene glycol and calcium oxide).

Keywords: Ethanol, Bio-ethanol, Fermentation, Microwave pretreatment, Distillation under vacuum , Dehydration.

1. INTRODUCTION

Renewable energy has been highlighted in the last three decades due to its potential to replace fossil fuel energy especially for transportation. Use of renewable energy sources, such as bio-ethanol and biodiesel as motor vehicle fuels, either pure or blended, have been growing rapidly for a variety of reasons. The turbulences in the oil market during the last decades and the global desire to reduce net CO_2 and other pollutants emission has made bio-ethanol a desirable fuel [1]. Sugars and grains are the dominant feed stocks for ethanol production, but lignocellulosic biomass is considered as the key feedstock in the future [2]. However, despite its high octane number, bio-ethanol contains only 67% of the energy content of gasoline, see table (1) [3].

Table (1) Physical and chemical properties of ethanol, methanol and gasoline [3]

Property		Methanol CH3OH	Ethanol C2H5OH	Gasoline C4-C12
Molecular weight (g/mol)		32	46	~114
Specific gravity		0.789 (298 K)	0.788 (298 K)	0.739 (288.5 K)
Vapor density rel. to air		1.10	1.59	33.0-4.0
Liquid density (g /cm3 at 298 K)		0.79	0.79	0.74
Boiling point (K)		338	351	300-518
Melting point (K)		175	129	-
Heat of evaporation (Btu/lb)		472	410	135
Heating value (kBTU/ gal)	Lower	58	74	111
	Higher	65	85	122
Tank design pressure (psig)		15	15	15
Viscosity (cp)		0.54	1.2	0.56
Flash point (K)		284	287	228
Flammability/ explosion limits	(%) Lower (LFL)	6.7	3.3	1.3
	(%) Upper (UFL)	36	19	7.6
Auto ignition temperature (K)		733	636	523-733
Solubility in H2O (%)		Miscib.(100%)	Miscib.(100%)	Negl. (_0.01)
Azeotrope with H2O		None	95% EtOH	Immiscible
Peak flame temperature (K)		2143	2193	2303
Minimum ignition energy in air (mJ)		0.14	0.23	-

The challenge, therefore, is to produce advanced bio-fuels that have high energy content and are compatible with storage and transportation infrastructures designed for petroleum based products, which are also economically feasible to produce on an industrial scale. Potential advanced bio-fuels that could supplement or replace gasoline include short-chain alcohols and alkanes [4].

Bio-ethanol is, presently, an important renewable source of liquid transportation fuel and offer many potential environmental and economic benefits. The production of the raw biomass material and its subsequent conversion to fuels creates local jobs, provides regional economic development, and can increase farm and forestry incomes. Bio-ethanol also offers many environmental benefits including reduction of carbon dioxide emissions associated with global climate change and improved waste utilization. The chemical composition of many bio-fuels, including bio-ethanol, also leads to improve engine performance and reduces unwanted pollutants such as carbon monoxide and unburned hydrocarbons therefore billions of litres of bio-ethanol are used annually as transportation fuels, and bio-diesel is gaining in popularity in some regions [5]. Microwave technology was used as a pre-treatment to reduce the disadvantage of anaerobic process and to improve volatile suspended solid (VSS) and chemical organic demand (COD) solubilisation by choosing three samples of local municipal wastewater treatment plants and exposed them to heating rate by domestic microwave oven. At 170°C, the VSS dissolution ratio of treated sludge reached 36.4% and COD dissolution ratio was about 25%. Under this typical hydrolysis parameter, microwave irradiation could shorten holding time to 5 min, compared to conventional processes that require more than 30 min [6]. Cysewski and Wilke [7] used vacuum technology to reduce the duration of fermentation process.

The fermentation process is followed by distillation and dehydration processes to reduce the water content of ethanol until 99.6% concentration. The distillation process produces alcohol concentration in the range 92-95%. Different techniques are available [8].

Ebraheem A. K. And Shahad H.A.K [9] used microwave pre-treatment to reduce the time of fermentation process of date syrup to produce bio ethanol.

2. EXPERMENTAL WORK

The aim of this research is to produce bio-ethanol from Iraqi dates crop with maximum possible concentration and minimum cost. The production procedure includes the following steps:

The date juice is pre-treated in a microwave to a specified temperature for a specified duration. Fermentation of the treated juice is then performed followed by distillation process under vacuum pressure which is then followed by dehydration process using two drying agents (ethylene glycol or calcium oxide) to the maximum possible concentration.

The Zahdi date species juice has been chosen due to its favourable properties **[9]** which are**:**

1. Its sugar content is 67.1%.
2. It has a beautiful golden colour.
3. One ton of Zahdi yields 0.7 ton of juice with 70% sugar concentration.

4. Its price is very cheap compared to other species.
5. Density of juice at a concentration of 70% is (1.300 to 1.363 kg/m^3).

2.1 Pre-Treatment and Fermentation Process.

Microwave technology is used as a pre-treatment to reduce the time of fermentation process of juice. Three samples of juice are exposed to heating in a domestic microwave oven at 140 °C for different residence periods {10 min, 20 min, 30 min}. It is found that when the residence period exceeds 30 min the sample will burn. After the pre-treatment the fermentation process is performed. Table (1) shows the materials and quantities used in the fermentation process for three samples. Same quantities are used for each sample.

Table (2) Materials and quantities used in the fermentation process

Material	Quantity
Juice	2 liters
distilled water	6 liters
The bread yeast	40 g

After the pre-treatment, the juice is mixed with other ingredients, shown in table (2), and the mixture is poured into the fermentation tank, fig (1).

1.iron tank, 2.special pump for chemicals, 3. cooling system, 4. pressure gauge, thermometer and vent valve, 5. volume flow meter, 6. plastic connecting pipe.

This process converts sugar into ethanol and carbon dioxide according to the following reaction:

$$C_6H_{12}O_6 \xrightarrow{\text{Yeast}} 2C_2H_5OH + 2CO_2$$

The fermentation procedure is as follows:

Six liters of distilled water is heated to 35 °C then the juice is added to the heated water and the mixture is put in the fermentation tank. The circulation pump is started without closing the fermentation tank until the ingredients (juice + water) are well mixed to form a homogeneous mixture at a temperature between 30-35 °C. This process continues for 4-5 hours with the tank open. The yeast is dissolved outside the tank in 0.5 L of distilled water which is at a temperature of 35 °C in order to stimulate the yeast. The dissolved yeast is added into the tank and the tank is closed tightly by the lid. The temperature of the ingredients must be maintained between 30-35 °C to ensure that the fermentation process continues to completion. The circulation pump is started to circulate the mixture and stimulate the yeast which speeds up the fermentation process. At the start of the fermentation process the temperature of the mixture rises and therefore cooling is needed to keep the temperature constant in the specified range. When the fermentation process is complete the output is known as "low wine" which contains 12% alcohol by volume.

2.2 The Distillation Process Under Vacuum Pressure

This process is designed to produce a bio-ethanol with a concentration of, at least 91%, from the low wine which contains 12% ethanol. The column utilizes the differences in the boiling points of ethanol and water to boil off and separate the ethanol. There are a large number of chemical compounds in the mixture of fermented juice and the disparity in degrees of boiling of these substances plays a crucial role in the separation of ethanol.

The distillation process is carried out in three stages to separate the bio-ethanol from heavy materials, yeast and impurities in the distillation column shown in fig (2) which is fabricated in the laboratories of Mechanical Engineering Department /Babylon University . During the distillation process the alcohol concentration is raised from 12% to 91% in three stages. During the first stage, the alcohol concentration is raised from 12% to 44%, while during the second stage the concentration increased from 44% to 81% and the output of the final stage is 91%.

The effect of prevailing pressure in the column on the produced alcohol concentration and the duration of the process is investigated. Tow vacuum pressures are tested, 0.7 bar vacuum and 0.5 bar vacuum.

The overall output of three stages is 100 ml of condensate with 91% bio-ethanol concentration at 20 ° C for each one litre of fermented juice.

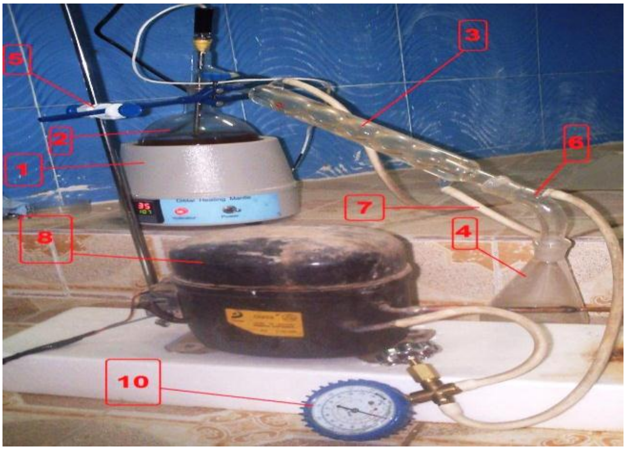

Fig (2) The Distillation Column

1. digital heating mantle, 2.flask capacity one litre, 3. condenser, 4. collecting flask, 5.condenser stand, 6. connections to connect different parts of the column, 7. cooling water supply, 8. Vacuum

pressure pumps, 9. water flow meter to measure cooling water flow rate, 10. vacuum pressure gauge.

2.3 The Dehydration Process Under Vacuum Pressure

This process is designed to produce ethanol at (99.6%) concentration. The alcohol with 91% concentration produced by the distillation process is dried under vacuum using two drying agents, either ethylene glycol or calcium oxide (CaO). Two vacuum pressures are tried either 0.7 or 0.5 bar vacuum.

The drying agent is mixed with the output of the distillation process for 10 min. The amount of drying agent equals 80% of the amount of distillate with 91% ethanol concentration. The mixture is then heated until evaporation in the dehydration column at a temperature lower than the evaporation temperature of the last stage of distillation process.

The remains of the dehydration process is heated to 157 ^{o}C to reduce its water content and cold to 80 ^{o}C and then recycled with the fresh charge of the dehydration column.

3. RESULTS AND DISCUSSION

3.1 Fermentation Process With Microwave Pre-treatment

The results show that the pretreatment reduces the fermentation time as shown in Fig (3). This reduction increases with residence time until 30 min after that any increase in residence time causes the sample to burn. The fig shows that 10, 20 and 30 min residence time reduces the fermentation time by 8.3%, 20.8% and 29% . The residence time can be increased beyond that if inert medium is used in the oven .

Fig (4) shows that the energy consumption per liter of ethanol in the fermentation process with microwave pre-treatment is greater than that in the fermentation process without microwave oven due to the energy consumed by the oven. Microwave pre-treatment reduces the time and effort required for the fermentation process. Power consumption in the microwave oven in present work is very high. It can be reduced by using the microwave oven which consumes less energy and preparing larger sample quantity.

3.2 Distillation Process Under Vacuum Pressure

The aim of the process is to study the effect of the prevailing pressure on distillation time and how much vacuum pressure is required. Two vacuum pressures are tried namely 0.7 and 0.5 bar.

Fig (5) shows the effect of vacuum pressure on alcohol concentration in the distillate. It is clear that as the vacuum pressure increases the evaporation temperature is reduced and the alcohol concentration is increased. However what is required for each stage is high alcohol concentration output and a shorter distillation time. It is found that the evaporation temperature of 62 °C under 0.7 bar vacuum gives 40% alcohol concentration and 1.35 hr distillation time and 70 °C evaporation temperature under 0.5 bar vacuum gives 41% alcohol concentration and 1.40 hr distillation time. These conditions are found to give high alcohol concentration in the distillate and short distillation time compared to other tested conditions Therefore these temperatures give a good time with larger increase in alcohol concentration in the distillate. Therefore these temperatures are recommended for this stage.

Fig (6) shows the effect of vacuum pressure on alcohol concentration in the distillate for the second stage. As in the first stage it is noticed that increasing the vacuum pressure reduces the evaporation temperature and increase the alcohol concentration. However it is clear form results that the temperature of 56 °C under 0.7 bar vacuum gives 80% alcohol concentration and 3.30 hr distillation time and 66 °C under 0.5 bar vacuum gives 81% alcohol concentration and 3.45 hr distillation time. Therefore these temperatures give a good time and good increasing in concentration. Therefore these temperatures are recommended for this stage.

Fig (7) shows the effect of vacuum pressure on alcohol concentration in the distillate for the third stage. As in the first and second stages it is noticed that increasing the vacuum pressure reduces the evaporation temperature and increases alcohol concentration. The results show that the 52° C under 0.7 bar vacuum gives 90% alcohol concentration and 5.45 hr distillation time and 63 °C under 0.5 bar vacuum gives 91% alcohol concentration and 6 hr distillation time. Therefore these temperatures give a good time and good increasing in concentration. Any further increase in temperature gives large reduction in concentration and small reduction in time which is not preferred. Therefore these temperatures are recommended for this stage.

The variation of alcohol concentration with evaporation temperature obtained in the present work is compared with the work of Ref [7]. As shown in fig (8) good agreement is obtained. When vacuum pressure increases the evaporation temperature is reduced and the alcohol concentration and distillation time are increased because little quantity of water will evaporate.

Fig (9) shows that the required time for distillation process is shorter in case of vacuum pressure than under atmospheric pressure. As the vacuum pressure increases the total distillation time decreases due to higher rate of evaporation. However increasing the vacuum pressure from 0.5 to 0.7 bar vacuum given small reduction in distillation time which is insignificant and therefore no need to reduce the vacuum pressure more than 0.5 bar.

Fig (10) shows that the energy consumed in the process of distillation at atmospheric pressure is less than the energy consumed in process under vacuum pressure because more power consumption is consumed in the vacuum pumps. Although process under vacuum pressure has shorter time. Power consumption in the vacuum pump in present work is very high and it can be reduced by using the vacuum pump consumes less energy.

3.3 Dehydration Process Under Vacuum Pressure

The aim of the process is to study the effect of prevailing pressure on alcohol concentration and dehydration time and how much vacuum pressure is required. Two experiments are done one under a vacuum pressure of 0.7 bar while the other under a vacuum pressure of 0.5 bar.

Fig (11) shows the results of this experimental of using ethylene glycol as drying agent. It is found that the evaporation temperature of 51 °C under 0.7 bar vacuum and reduction in time about

25%compared to atmospheric pressure and 64 °C under 0.5 bar vacuum and reduction in time about 12.5%compared to atmospheric pressure. These temperatures give a good time with large increase in alcohol concentration. Therefore these temperatures are recommended for this process.

Fig (12) shows the results of this experimental of using CaO as drying agent. It is found that the evaporation temperature of 48 °C under 0.7 bar vacuum and reduction in time about 34%compared to atmospheric pressure and 60 °C under 0.5 bar vacuum and reduction in time about20%compared to atmospheric pressure. These temperatures give a good time with a large increase in alcohol concentration. Therefore these temperatures are recommended for this process.

Figs (13 &14) show that the required time for dehydration process is shorter in case of vacuum pressure with CaO than other cases.

The results show that the energy consumption in the dehydration process under atmospheric pressure with CaO less than other methods because more power consumption is consumed in the vacuum pumps. Although process under vacuum pressure has shorter time. Power consumption in the vacuum pump in present work is very high and it may be reduced by using the vacuum pump which consumes less energy.

4. CONCLUSIONS

From the results of the present work, the following conclusions can be written:-

1- Date crop is an important raw material for the production of biofuel (ethanol).

2- Use of microwave technology for pre-treatment of the juice reduces the time required for the fermentation process by 30%.

3- Distillation process under vacuum pressure requires shorter time compared to atmospheric pressure.

4- Dehydration process under vacuum pressure with CaO is best from other processes because this process gives high concentration and reduction in time about 34% compared to atmospheric pressure.

5- Calcium Oxide (CaO) gives good results in terms of high concentration of ethanol and less cooling water and good reduction in time under vacuum pressure in dehydration process.

6- The total cost to produce one liter of ethanol is 1021 ID which is about 85 cents. It is found that 1 ton of Zahdi yields 300 L of ethanol.

الخلاصة:

يمكن إنتاج الايثانول الحيوي من محاصيل زراعية مختلفة مثل قصب السكر، البنجر السكري ،التمور الخ. من المعروف أن العراق يمتاز بإنتاجه الوفير من التمور بأنواعها المختلفة. من خلال البحوث التي أجريت من قبل عدد من الباحثين وجد أن صنف تمر الزهدي يمتاز بمحتواه العالي من السكر مقارنة مع الصنوف الأخرى وكذلك بوفرة إنتاجه ورخص ثمنه. لذلك تم استخدامه في هذا البحث لإنتاج الايثانول الحيوي. تم في هذا البحث دراسة تحسين عملية الإنتاج من حيث الكمية النتيجة والكلفة باستخدام تقنيتين جديدتين.

تتكون عملية إنتاج الايثانول الحيوي الاعتيادية من التمر من عدة مراحل هي إنتاج الدبس، التخمير، التقطير وأخيرا التجفيف. يتم إجراء هذه المراحل تحت ظروف الضغط الجوي. ولغرض زيادة الكمية المنتجة.

لوحدة الكتلة من التمر تم اختبار تقنيتين جديدتين. في التقنية الأولى تم إضافة مرحلة جديدة قبل مرحلة التخمير وهي مرحلة تسخين نموذج الدبس في فرن الموجات الدقيقة لفترة محددة ومن ثم إجراء العمليات اللاحقة. أجريت عملية التسخين عند درجة حرارة $140\ ^oC$ لثلاث فترات زمنية هي 10, 20, 30 min

والغرض من هذه المرحلة الجديدة هو تقليل زمن مرحلة التخمير. وجد من التجارب انه عند زيادة فترة التسخين (زمن الاستبقاء) أكثر من 30 min سوف يحترق النموذج في الفرن. أما التقنية الثانية فهي اجراء مرحلتي التقطير والتجفيف تحت ضغط اقل من الضغط الجوي (تخلخل الضغط) والغرض من هذه هو تقليل درجة حرارة تبخر الماء وبذلك يمكن تقليل كلفة الطاقة وكلفة الإنتاج. تم إجراء هذا الاختبار عند ضغط فراغ 0.7 bar, 0.5.

بينت نتائج الدراسة أن تسخين الدبس في الفرن يقلل زمن التخمير بنسب 8.3, 20.8 and 29% لفترات التسخين 10, 20, 30 min على التوالي. أن هذا يؤدي إلى تقليل الطاقة المستهلكة خلال مرحلة التخمير وبالنتيجة تقليل كلفة الإنتاج. كذلك بينت النتائج أن إجراء مرحلة التقطير تحت ضغط مخلخل يقلل الزمن اللازم لإكمال مرحلة التقطير بنسبة 32% وكلما قل الضغط قل الزمن. أما مرحلة التجفيف وهي الأهم لكونها تستغرق الوقت الأطول لإنتاج الايثانول بالتركيز المطلوب فقد بينت نتائج الدراسة أن إجراءها تحت ضغط مخلخل يقلل الزمن بنسبة 33%. تم اجراء التجفيف باستخدام احدى المادتين التاليتين وهما calcium oxide أو ethylene glycol .

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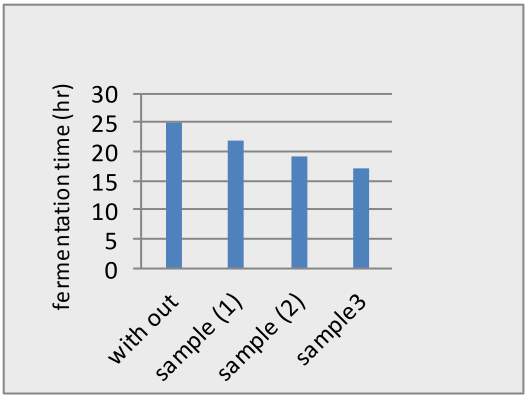

Fig (3) Total fermentation time for each sample

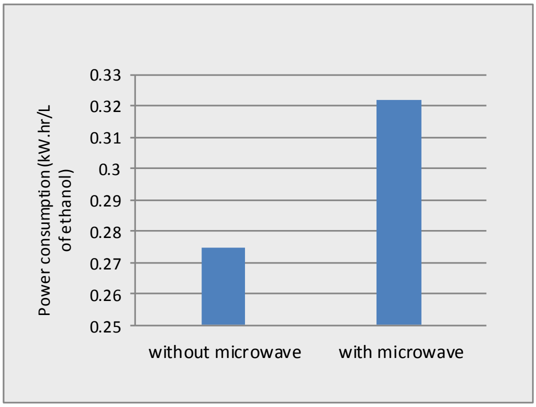

Fig (4) A comparison of power consumption during fermentation process for both techniques

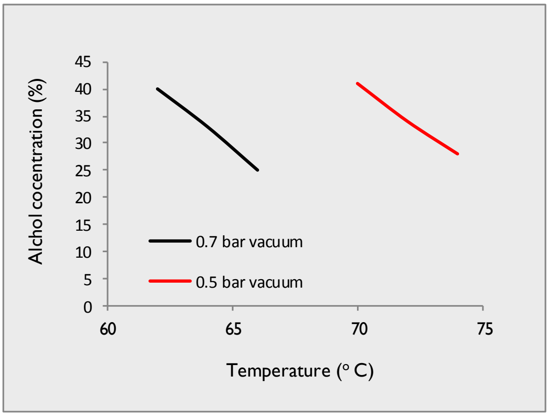

Fig (5) Effect of evaporation temperature on alcohol concentration under different prevailing pressures for the first stage of distillation

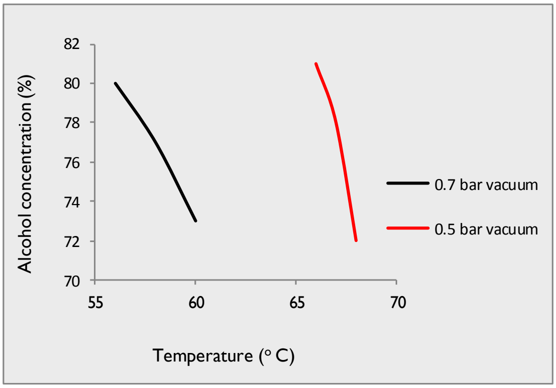

Fig (6) Effect of evaporation temperature on alcohol concentration under different prevailing pressures for the second stage of distillation

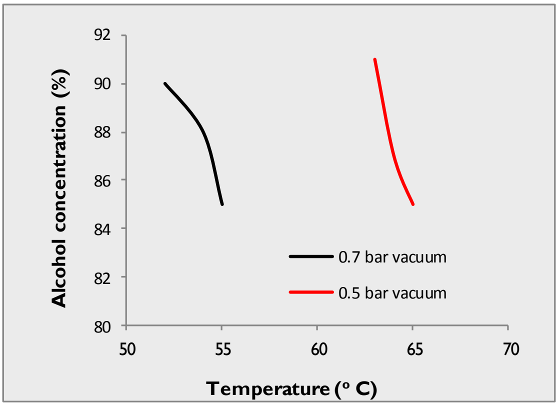

Fig (7) Effect of evaporation temperature on alcohol concentration under different prevailing pressures for the third stage of distillation

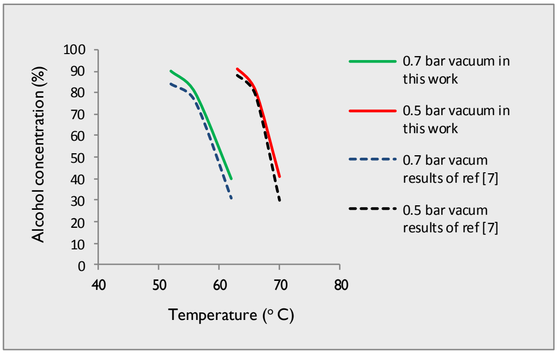

Fig (8) A comparison of present work results with results of ref [7]

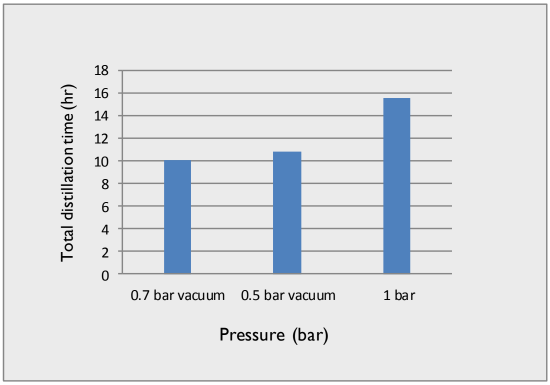

Fig (9) Total distillation time for each pressure

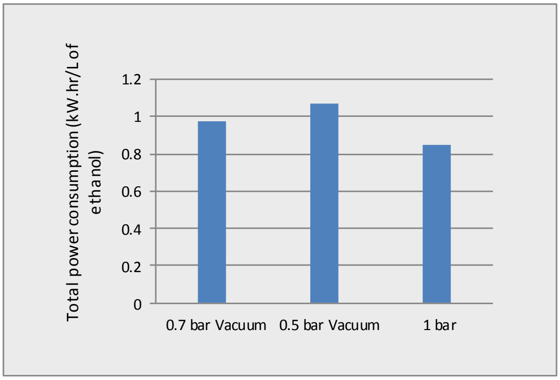

Fig (10) Effect of prevailing pressure on total power consumption in distillation process

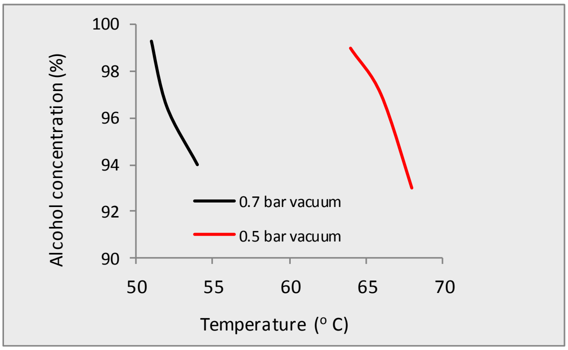

Fig (11) Effect of evaporation temperature and prevailing pressure on alcohol concentration for the dehydration process with ethylene glycol

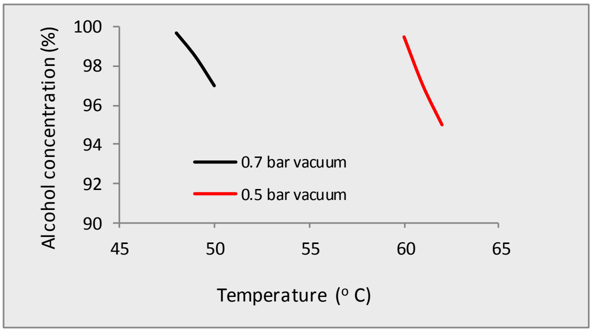

Fig (12) Effect of evaporation temperature and prevailing pressure on alcohol concentration for the dehydration process with CaO

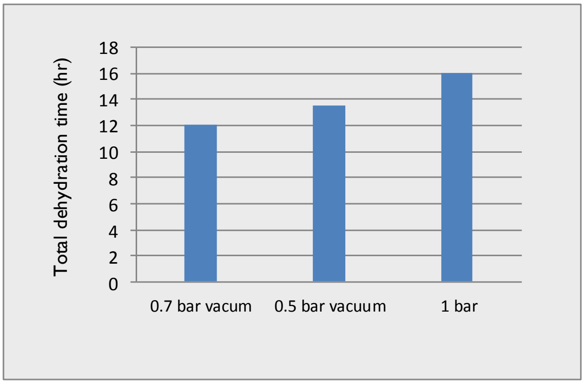

Fig (13) Total dehydration time for each pressure with ethylene glycol

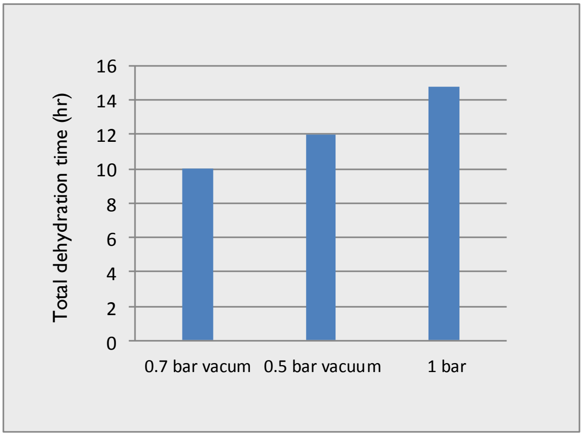

Fig (14) Total dehydration time for each pressure with CaO