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Bioethanol Production from Wastes: An Experimental Evaluating Study for Iran

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ABSTRACT

Energy crisis in the world motivates countries to hire new and renewable energies. One of the main and valuable renewable sources of energy is agricultural waste. This is widely disposed of through the world during the harvest, packing, and transportation. In many countries, agricultural waste is considerably weighty. Nonetheless, most of that is used for animal feed or herbal fertilizer and no useful value is added. Despite its location in an arid region, Iran produces various citrus, cereals, and vegetables in high tonnage. The waste of the agricultural product, especially those disposed of by the food processing industries, such as fruit juice factories, remains also useless. The potential of the residues to extract biofuel is investigated in the current experimental study. Six samples of abundant agricultural products in Iran are chosen: sugarcane, grape, potato, orange peel, date, and mulberry. The processes of pretreatment, hydrolysis, and fermentation are performed and the extracted juice is directed to the distiller to gather bioethanol. To evaluate the distilled juice purity, a gas chromatography test is carried out. It is shown that date and mulberry can produce a maximum of 29.5 and 23 ml (ethanol)/100 g (dry waste) as the most efficient agricultural products.

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1. INTRODUCTION

Drastic increment in energy demand in the current century motivates humans to extract fuel from new sources. Renewable energies such as biofuels are the most prominent targets to subside fossil fuel dependency for countries. Utilizing biofuels takes countries to the safe side regarding oil price fluctuating, air pollutions, and fossil fuel depletion [1]. Among the threats of applying fossil fuels, outdoor pollution, causing more than 4.2 million deaths annually [2], motivates the idea of finding alternative fuels. Extracting fuels from crop wastes or as a by-product during the food processing of agricultural products is so promising to achieve sustainable energy development. Among different types of biofuels, bioethanol is more common due to source variety and its ability to blend with fossil fuels in commercial vehicles [3,4]. Although it has a lower energy content than gasoline, its octane number is higher, which preserves the motor from knocking in the condition of high pressure [5]. Further, higher oxygen content and non-toxic combustion products lower air pollution.

Bioethanol production in Iran is far behind some other countries, owing to the high resources of crude oil and natural gas [6]. However, during pandemic Covid-19, there emerges a high demand for disinfection liquids, especially ethanol, which can be supplied through bioprocessing. Switching from fossil fuels to biofuels and enhancing the economic value of agricultural products by considering the waste is potentially a promising step to develop a country. It is estimated that more than 21.5 million tons of agro-food waste are annually disposed of in Iran, which can produce more than 5.5 billion liters of bioethanol [7]. The lignocellulosic feedstock and sugar or sucrose ingredient in the gardening products, which can be found most abundantly in Iran's crops, are two main sources of producing bioethanol that can be investigated in the current experimental study. Bioethanol, which is the most produced biofuel by up to 95 % of the global production, can be replaced by 32 % of the global gasoline consumption [8]. This means the creation of economic interest from something with no profit. The reduction of emission in dirty cities can be another indirect economical key that should be considered as an achievement. However, the economic chain of biofuel production in Iran should be planned and the policies should be accurately passed, as well. Although this study focuses on Iran as its case study, the goal of this study is inclusive of the case in other countries where bioethanol by-producing has been developed and, therefore, some published works have been found in the literature.

Sritrakul et al. [9] investigated bioethanol formation from sugarcane pith using two processes of simultaneous saccharification and fermentation as well as separate hydrolysis and fermentation. Their case study was Thailand. The former was found more efficient than the latter. Maximum bioethanol extraction was achieved by 3.7 g/L. Zakir et al. [10] compared ethanol production from sugarcane bagasse in terms of various variables affecting enzymatic hydrolysis and yeast fermentation process. Their study showed the optimum value of temperature and PH for the process of production. The fermentation process remained unchanged after a certain time, as the concentration of ethanol

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in water was kept constant. Moodley and Kana [11] evaluated the bioethanol production from sugarcane leaf and optimized the involved variables in the hydrolysis process. There was no difference between enzyme filtration and infiltration on the production rate.

Noufal et al. [12] extracted bioethanol from the flour of the potato waste. After fermentation of hydrolysate with Saccharomyces cerevisiae, the maximum ethanol rate of 33 g/L was given. The best temperature of the fermentation process was at 35 °C with the optimum pH between 5 and 6. Hashem and Darwish [13] showed that bioethanol production from potato wastes could be obtained without adding organic additives, while some minerals such as Zn were required in a small concentration. The maximum ethanol production rate of 5.8 g/L was achieved. Yamada et al. [14] found that ethanol production by enzyme hydrolyses of potato peel could be improved by a factor of 2.5 if the potato mash with an equal weight ratio was added.

In a case study for Bangladesh, Swaraz et al. [15] investigated bioethanol production from the wild date palm. They reached 0.278 g/g bioethanol after a batch fermentation of *Saccharomyces cerevisiae*. They emphasized the importance of dates as a promising sugary feedstock for ensuring enough food and energy. Using a low-cost and direct bioethanol production from the date palm, Ben Atitallah et al. [16] introduced Wickerhamomyces anomalus X19 yeast and extracted a maximum of 61.5 g/L ethanol by a batch mode fermentation. Corbin et al. [17] utilized white and red grape marcs to evaluate the ethanol product. Using sulfuric acid pretreatment, about 10 percent of glucose liberation after saccharification was reported. Then, 270 L/tone bioethanol extraction was achieved. Bioethanol production from grape pomace was evaluated by Rodriguez et al. [18] using solid-state fermentation. The optimum time to fulfill the fermentation process was detected. Oberio et al. [19] optimized the parameters involved in hydrolysis and fermentation of orange peel. Two steps of hydrolysis were taken. They showed that the hydrolysis could be managed such that the fermentation process would become unimportant. Joshi et al. [20] reported bioethanol and biobutanol production from orange peel using Saccharomyces cerevisiae and Clostridium acetobutylicum. A maximum value of 4.1 g/100mL bioethanol was extracted after hydrolysis and fermentation, while 19.5 g/L bioethanol was given without adding nutritional supplements. Although the berries can be an appreciated source of bioethanol production, bioethanol extraction from mulberries has been rarely reported. This is, however, one of the goals of the current study. The work of Wang et al. [21], as the only publication on this issue up to now, presented Fed-nonisothermal-simultaneous saccharification to achieve the goal of higher bioethanol production from mulberry. Ethanol was extracted by about 63.9 g/L, improving more than 30 % in comparison with those traditional processes. Pretreatment using H_3PO_4/H_2O_2 was introduced as the most effective method.

The current work to evaluate bioethanol extraction from some abundant agricultural production in Iran. Pretreatment, hydrolysis, and fermentation are followed to prepare the juice added to the distiller. Gas Chromatography (GC) test is carried out to measure the purity of the obtained ethanol. This study shows the potential of crop waste in Iran to add economic value and produce cleaner fuels.

2. MATERIALS AND METHODS

2.1. Raw materials

The raw materials are chosen from the most seeded crops in Iran. According to the FAO (Food and Agriculture Organization of the United Nations) report [22], Iran is one of the most important countries to produce some kind of agricultural products relating to human food. Iran can compete with many countries in production date, grape, orange, sugarcane, potato, and mulberry, which are considered as the sources of bioethanol extraction in this study. Due to the poor and traditional harvest as well as non-advanced food industries, the waste of these crops is considerable. Table 1 shows the details of the chemical compositions of the raw materials applied. The presented values may contain ±1 % deviation, in maximum.

It should be noted that in this table, Dates 1 to 3 belong to Jiroft, Bushehr, and Hajiabad (located near Bandarabbas city) cities, correspondingly. The sugarcane was supplied from Haft Tappeh of Shush city. Other crops were provided from Kashan and its countryside. Dried mulberries (Morus alba) were used for the tests.

Table 1. the composition weight percentage of the raw materials used in this study

Material	Raw material					Moisture
	Carbohydrates	Cellulose	Hemicellulose	Lignin	Ash	
Date 1	80.45	5.70	3.9	1.6	1.78	20
Date 2	82.43	6.50	2.3	1.3	1.89	12
Date 3	76.47	5.25	2.9	1.0	1.55	14
Sugarcane	95.03	21.3	18.4	5.4	2.84	13
Orange peel	38	13.61	6.10	2.10	1.5	75.4
mulberry	75.30	9.68	4.22	2.88	3.99	11
grapes	79.20	17.87	7.0	10.23	4.65	3.3
Potato	17.88	0.55	0.045	0.20	0.29	70.5

2.2. Pretreatment, hydrolysis, and fermentation

Figure 1 shows a schematic view of bioethanol extraction from target agricultural products. The process is presented in Figure 2 with more details using the block box diagram. The wastes are collected and washed without adding any chemical substance. The raw pomaces can be found in the farms or residue of the food processing industrial units. Then, all of the wastes are crushed and chopped up on a similar 5-2 mm scale, depending on the initial size of the materials [12-14]. The pretreatment is followed by stirring the mixture at a temperature of 100 °C for about 25 minutes [23]. The

obtained juice is injected into a fermentation container where the liquid is kept for 48-72 hours depending on the product type. Saccharomyces cerevisiae, made by Iran Malas Company [24], is used as the yeast. The used yeast has a lower protein rate than 45 % and lower phosphor and ash rates than 6 % and 3 %, respectively, according to its brochure. The number of 2×1010 living cells is found per gram. The concertation of the yeast should be 5 g per 1 kg of the substrate [23]. By achieving better performance, the yeast is grown by solving problems in a mixture of distilled water and sugar at a temperature of 50 °C. The fermentation process is kept at a nearly constant temperature of 30 °C in anaerobic conditions [25-27]. Therefore, the batch container should be sealed and only a scape way for carbon dioxide, called an airlock (see Figure 2a), is implemented. Figure 2b shows the carbon dioxide produced in the fermentation process, as the calcium carbonate-water mixture became darker. The extracted liquid at the end of the fermentation process is filtered and collected. The filter should be used on a fine scale to remove all the solid particles. It is worth noting that all the processes were performed in the city located at about 940 meters over the sea level at the room temperature of 19±1 °C.

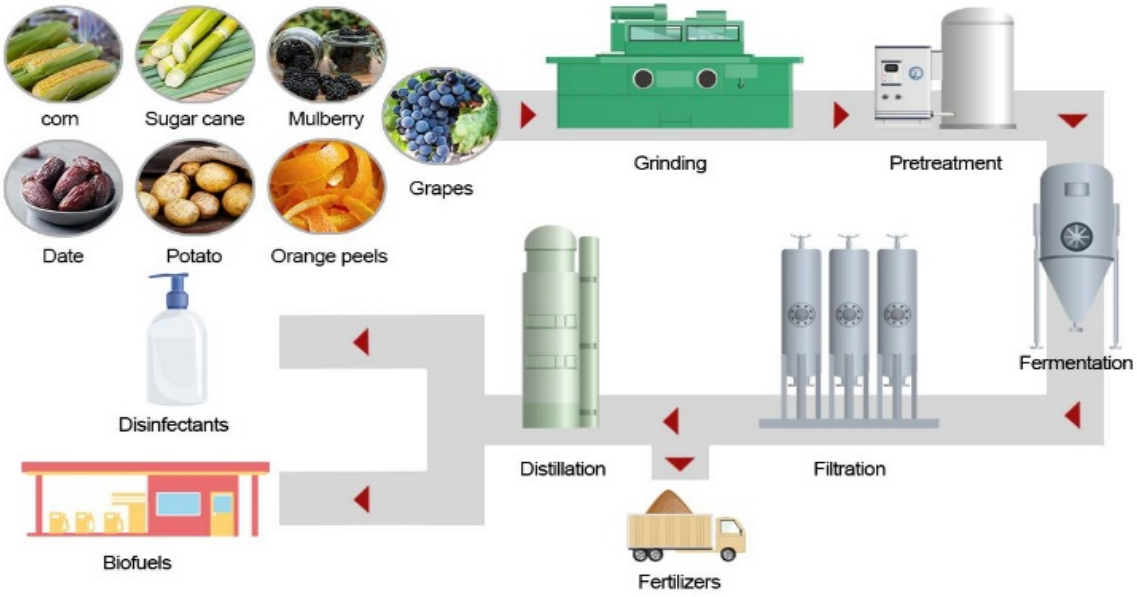

Figure 1. Schematic bioethanol production process

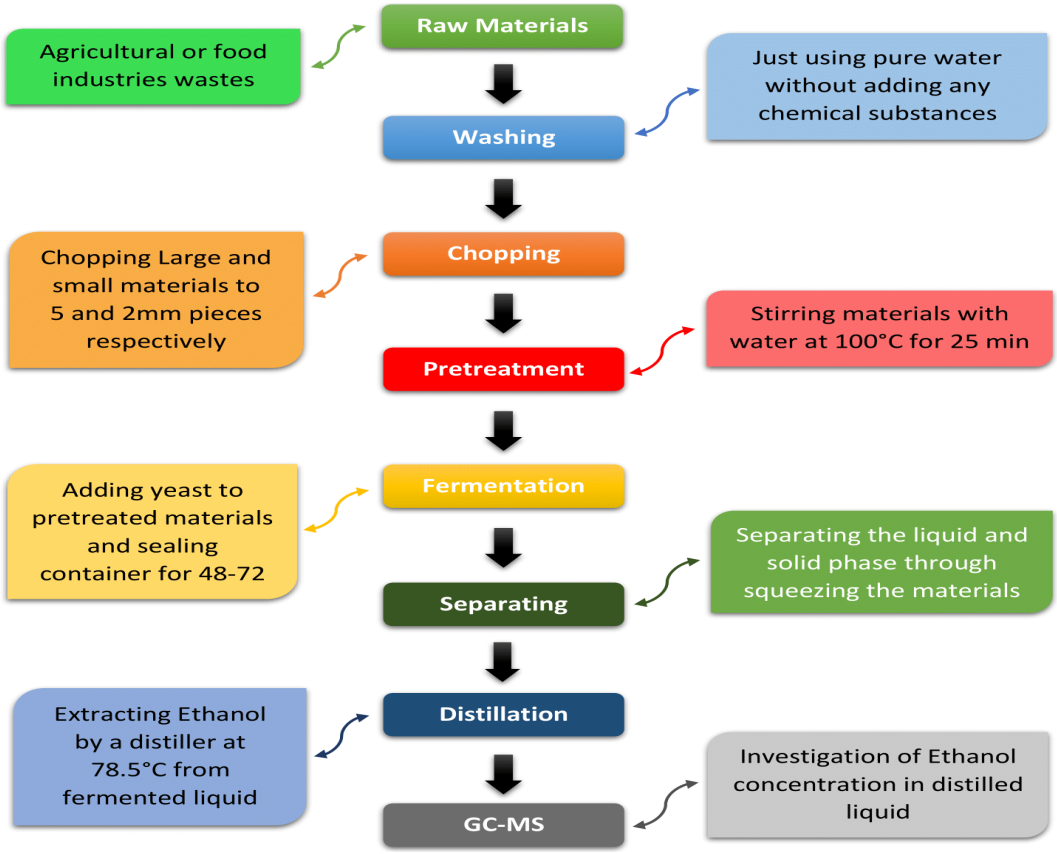

Figure 2. Block diagram of the production process

2.3. Distillation and gas chromatography

The filtered juice is subsequently added to the distiller (see Figure 4) and the ethanol is extracted at a temperature of 78.5 °C. The temperature is set at a certain value by a digital thermometer made by OMEGA Company. To verify the purity of the alcohol, a sample is tested in a GC machine (Agilent 6890: Mass selective detector 5973N) made in the USA. It contains a non-polar column (HP-5MS) with a length of 30 m and an inside diameter of 0.25 mm. Helium is used as the carrying gas. The GC machine injection volume is 0.5 µl with a split ratio of 50. For a test process, the Column temperature was initially maintained at 40 °C for 5 minutes. It was then heated to the temperature of 60 °C with a rate of 2 °C/min and finally, it was allowed to become hot at a temperature of 150 °C at a rate of 4 °C/min. The lab temperature during the tests was kept at 20±1 °C. The outputs are displayed in the next main section. The produced ethanol can be utilized as the biofuel source or disinfector.

2.4. Uncertainty and statistical analysis

Any experiments include bias and random errors [28]. Bias or systematic error is related to the measurement machines and can be found from the catalogs. GC machine is the main unit in the current study, having the bias error, called b_1, lower than 0.1 %, depending on what type of material is used. However, the upper limit is chosen to calculate the total uncertainty, here. The thermometer of the distiller could be another source, which was the precise one made by OMEGA company with the disparity rate lower than 0.2 °C, meaning lower 0.2 % error, as b_2. The error of weight measurement with the scales was about 1 %, denoted by b_3.

Random error, as the other source, derives from any unsteadiness or chance occurrence that may be unavoidable during the tests. To subside this source of error, we repeated each test five times and reported the arithmetic average of the data obtained as the result. Standard deviation, SD, for each test case was fallen in the range of 1-2 % of the arithmetic average value. Total uncertainty, TU, which reads [28]:

$$TU = \sqrt{b_1^2 + b_2^2 + b_3^2 + SD^2} \quad (1)$$

is provided as the error bars in the final absolute value of the ethanol extracted (See Figure 8). This is calculated by about ±2.3 %, in maximum. It should be noted that following variance analysis using ANOVA, null hypothesis was refused at a significance level of $P<0.05$.

(a) **(b)**

Figure 3. (a) The airlock and (b) the carbon dioxide produced

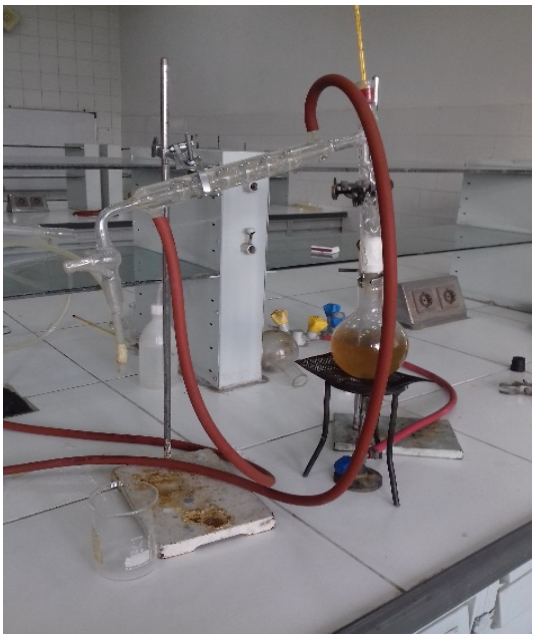

Figure 4. The distiller unit in the lab

3. RESULTS AND DISCUSSION

The GC results for sugarcane and potato are presented in Figure 5. This indicates that ethanol is the dominant species found in the sample. The value of ethanol for these two samples is nearly equal. However, the potato includes more undesirable chemical substances with a higher boiling point than the ethanol that may affect the engine performance, which requires to be purified. However, the higher flash points of 2-Methyle 1-Propanol and 3-Methyle 1-Buthanol are 29 °C and 42 °C compared to those of ethanol, as 12 °C, which can contribute to greater safety of the combustive system. Acetic acid is also reported for the potato sample, which causes a weak aqua-acidic environment after solving in the water of the combustion products.

Figure 6 depicts the pie charts of the GC test for three types of Iranian dates. The main species of the distilling process is ethanol and Date 2 contains a higher ethanol concentration. This is followed by Dates 3 and 1. The second dominant species for three samples is 3-Methyle 1-Buthanol, similar to previous results for sugarcane and potato. The high sugar content of the data can be converted into ethanol during the fermentation process. However, the optimization for temperature and other conditions for the batch container is substantially beneficial.

The results for grape, orange peel, and mulberry are presented in Figure 7. They can be sorted for ethanol production by ordering as stated earlier. The side products following the fermentation of mulberries are more significant. It can be implied that a longer period of hydrolysis is required with alternative methods. Further, the importance of purification is highlighted. Similar to the other cases, this figure shows that 3-Methyle 1-Buthanol is the second-highest concentration in the mixture.

The mass of ethanol extracted by each sample is given in Figure 8. To determine the value of concentration in terms of a milliliter bioethanol produced for 100 g of dry material, the following relation can be used.

$$C = \frac{L_D(g) \times S_E}{100 \times M_t(g) \times \rho_E(\frac{g}{ml})}, \quad (2)$$

where C is the concentration, L_D the mass of distilled liquid, ρ_E the ethanol density, M_t the total solid weight, and S_E the mass fraction of the ethanol. L_D and M_t can be easily weighted and S_E is provided by the GC machine.

Figure 8 shows that Date 2 can produce 29.5 ml ethanol for 100 g dry material, more than another agricultural residue, indicating that Bushehr date contains the higher value of ethanol among the date types examined. Date 1, i.e., the crop of Jiroft, is set in the next step by assigning 27 and 23 ml ethanol to 100 g dry material, respectively. Despite high sugar content, sugarcane falls in the last position. It is shown that the sugarcane samples should be processed by optimizing the pretreatment, hydrolysis, and fermentation conditions. It can be concluded that the waste of dates largely disposed of in Iran is a substantial source of bioethanol production, deserving greater attention to exploitation and extensive development.

The current study aims to accentuate the high potential of biofuel production in Iran. Therefore, the absolute value of the bioethanol production may be higher [18, 21, 29] or lower [9, 15, 30] than other similar attempts found in the literature, perhaps, in part, due to the different pretreatment and fermentation processes. If the involved variables such as time, temperature, and pH of the pre-test process become optimized, higher production may be attained. Further, the raw material of the tests is one of the other sources of probable disparity of the results compared to those earlier published. The compositions of the crops are different, depending on the soil, weather, and plant breeding conditions [31, 32].

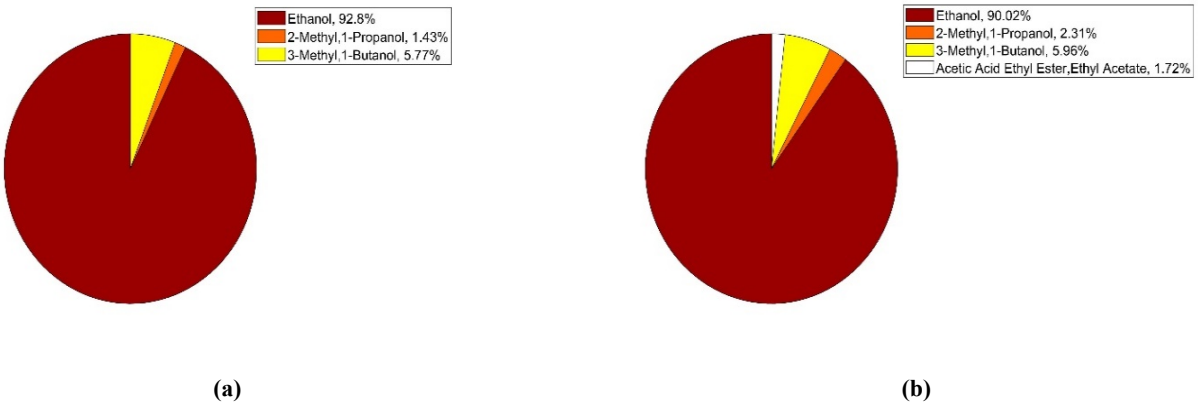

Figure 5. The results of the GC test for (a) sugarcane and (b) potato

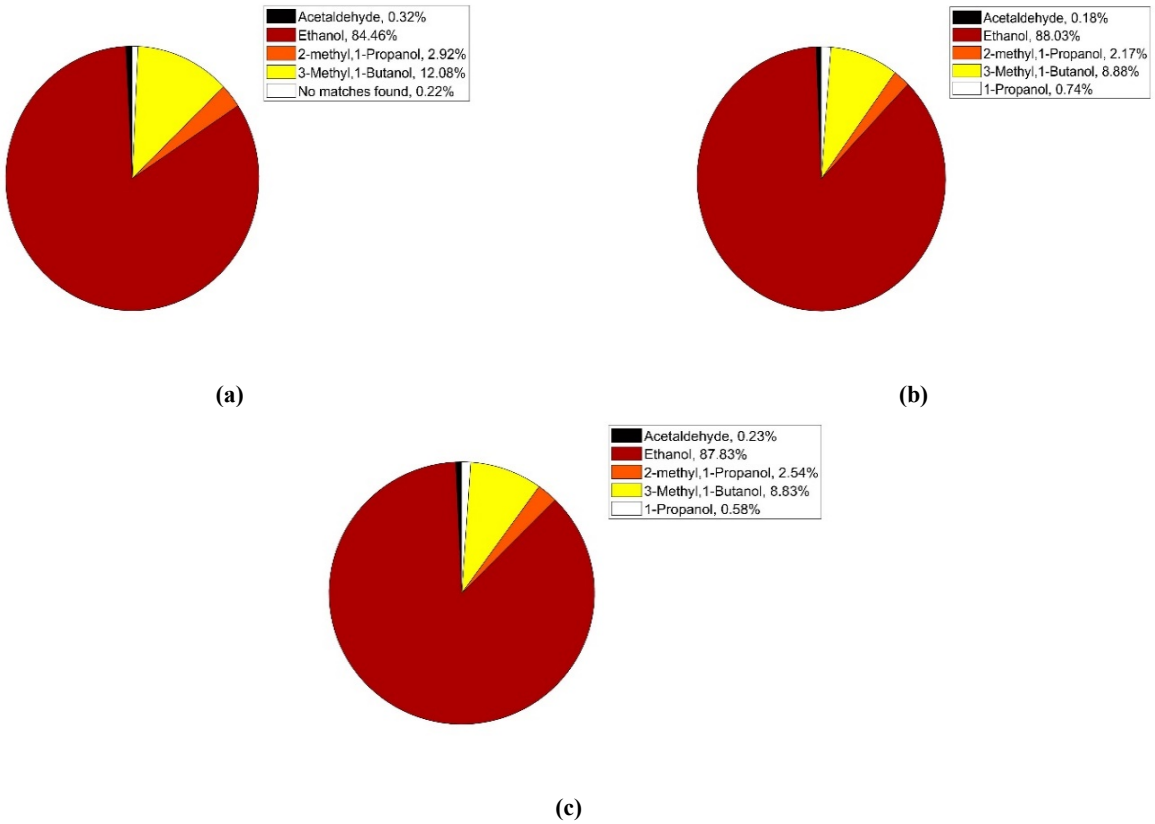

Figure 6. The results of GC test for (a) Date 1, (b) Date 2, and (c) Date 3

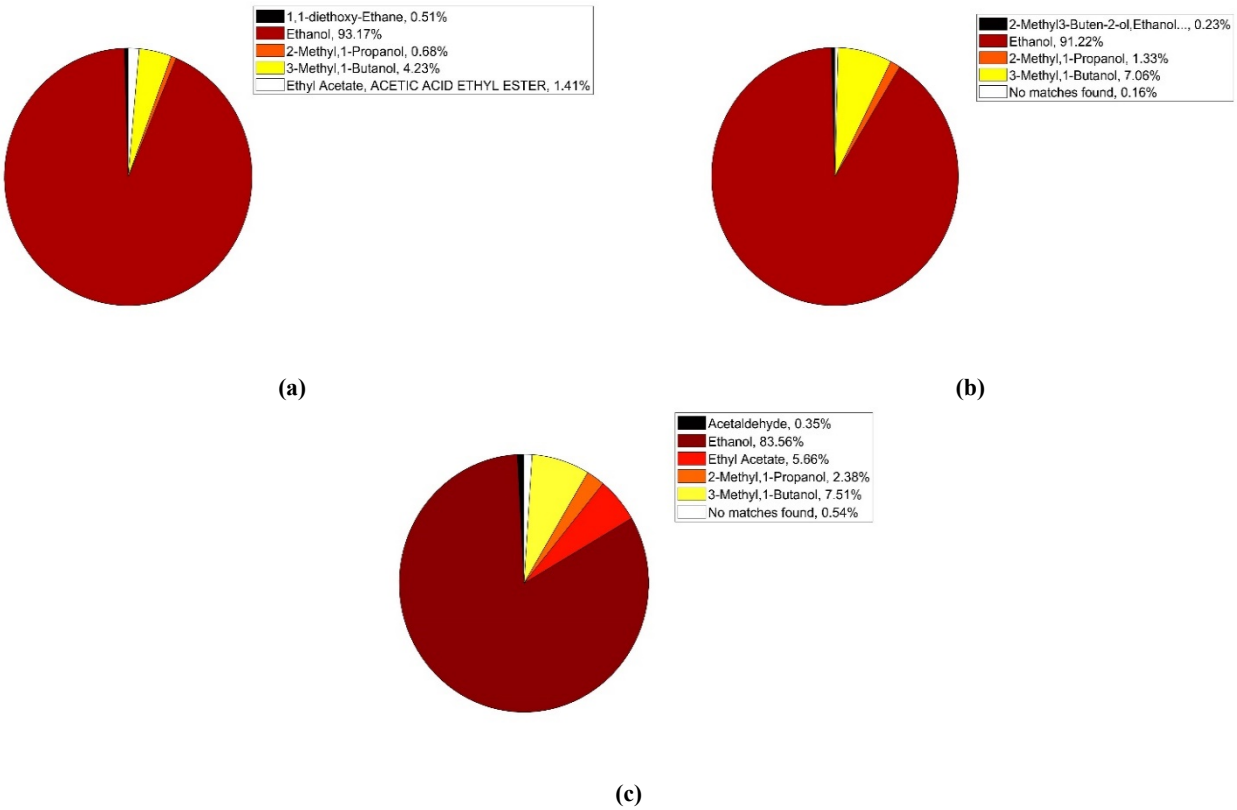

Figure 7. The results of GC test for (a) grape, (b) orange peel, and (c) mulberry

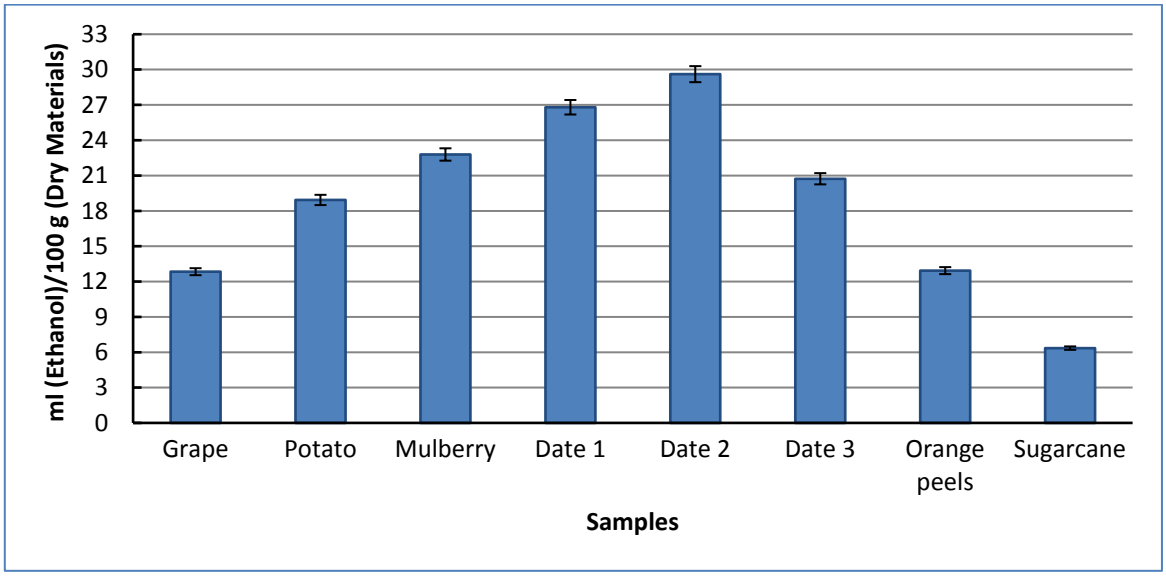

Figure 8. The volume concentration of ethanol extracted by each sample

4. CONCLUSIONS

The significance of biofuel in the current energy situation of the world cannot be neglected. Many countries seek new sources of energy, which are secure and independent of economical fluctuations. The waste of agricultural products, which is heavy in many countries due to wide food processing industries, can be considered as a promising energy source. Bioethanol is an applicable fuel in cars, in absolute or blended form. This can be produced by the agricultural residues, as studied experimentally in this work for Iran. The samples of many products in Iran, including sugarcane, potato, date, grape, orange peel, and mulberry, were gathered and the processes of pretreatment, hydrolysis, and fermentation were applied to them. The extracted juice was evaporated and condensed in the distiller to obtain the bioethanol. All samples showed an acceptable continent of ethanol. Bushehr date (29.5 ml (ethanol)/100 g (dry waste)), Jiroft date (27 ml (ethanol)/100 g (dry waste)), and mulberry (23 ml (ethanol)/100 g (dry waste)) were shown to be the most efficient products. The bioethanol production should be improved by optimizing the parameters involving the pretreatment and fermentation processes, which can be the subject of future study. Another type of fermentation process which can be more effective such as applying organic acids like formic, acetic, maleic, citric, and tartaric acid can be also considered as another development on the current work for future works. Further, many other agricultural wastes such as

cereals stalk can be examined as the source of bioethanol production in Iran. The current study showed that the biofuel industries should be set up and developed in Iran, ensuring energy saving and alleviating air pollution. This scheme could be potentially lucrative and beneficial for everybody who invests in this industry.

5. ACKNOWLEDGEMENT

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NOMENCLATURE

b1	GC machine bias error
b2	Thermometer bias error
b3	Weight measurement error
C	Concentration
L_D	Mass of distilled liquid
ρ_E	Ethanol density
M_t	Total solid weight
S_E	Mass fraction of the ethanol
Abbreviation	
GC	Gas Chromatography
SD	Standard Deviation
TU	Total Uncertainty

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