STM JOURNALS

Journal of
Alternate Energy
Sources & Technologies

ISSN: 2230-7982 (Online)
ISSN: 2321-5186 (Print)
Volume 15, Issue 3, 2024
September–December
DOI (Journal): 10.37591/JoAEST

https://journals.stmjournals.com/joaest

Research JoAEST

Production of Bio-Ethanol from Mixed Fruit Pulp

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Abstract

Speedy depletion and the continuously increasing cost of fossil fuels, is forcing the world to switch to renewable energy and perennial fuels. Among the different sources of renewable energy, bio-ethanol appears as a potential bio-fuel, which could be produced by a variety of biomasses available in abundance. This article mainly focuses on the utilization of completely waste mixed fruit pulp for bio-ethanol production. Bio-ethanol was extract from waste mixed fruit pulp, using acid hydrolysis followed by distillation. Experiments were carried out in controlled conditions of temperature and concentrations of sulphuric acid. Lab experiments indicate that optimum hydrolysis conditions for the maximum production of bio-ethanol from the mixed fruit pulp should be 35% of H_2SO_4 concentration manintaining at 70°C temperature. At these conditions, 560 mL bio-ethanol from per kg of waste mixed fruit pulp could be produced. The energy content of the mixed fruit pulp bio-ethanol was 21.54 MJ/kg. Finding of this research explores an effective utilization of waste fruit pulp to get liquid bio-fuel which may help to meet the challenges and unremitting increasing demand of energy.

Keywords: Bio-ethanol, fruit pulp, waste biomass, hydrolysis, distillation

INTRODUCTION

Globaly energy demand is rising day by day due to the growing economy, increasing population, exponential urbanization, technological advancements, and rising power spending rate [1]. On the other hand, a major supplier of our growing energy need, ‘petroleum’ life is decreasing. With this concern bio-fuel like bio-ethanol is appearing as a promising fuel, which could be blended with petrol or may use as a single fuel in vehicles [2]. The use of bio-ethanol in transportation not only reduces the fossil fuel demand but also supports to balance CO_2 cycle and finally the greenhouse effect [3]. A number of biomasses could be used for the production of bio-ethanol; however, fruits/fruit pulp may be interesting feedstock for the production of bio-ethanol as it is very rich in reducing sugars.

India is the second-largest producer of fruits and vegetables in the world; however, only 1.8% of the total production of fruits is being utilized for processing. To reduce the waste of fruits, and to add value to them, a number of steps have been taken such as canning, dehydration, pickling, preservation and bottling have been introduced. However, the percentage of processing of fruits is less than 3% as compared to countries like China (23%), the United States (65%), and the Philippines (78%). The waste materials (plant by-products) are very rich in valuable compounds. It can be utilized in various industries as the novel, low-cost, economical, and natural sources of dietary fibber, antioxidants, pectin, enzymes, organic acids, food additives, essential oils, etc. through different methods of extractions, purifications, and fermentations [2, 4–6]. Many of the industrial fruit wastes have been exploited for biogas production to compensate for

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Received Date: September 05, 2024
Accepted Date: September 10, 2024
Published Date: September 20, 2024

Citation: R.N. Singh, Apoorva Singh, Neha Gour. Production of Bio-Ethanol from Mixed Fruit Pulp. Journal of Alternate Energy Sources & Technologies. 2024; 15(3): 55–61p.

electricity needs in industrial sectors. However, producing biogas from the fruit wastes may be a time-consuming process. Considering the limitation of biogas production from fruit wastes, the production of bio-ethanol could be a lucrative option. In this study mixed fruit pulp waste (mainly oranges, pomegranate, pineapple, citrus limetta, sapota, etc.) were taken for bio-ethanol production.

MATERIALS AND METHODS [7–9]

Collection of Raw Material

Waste mixed fruit pulp was procured from the local markets of Indore, Madhya Pradesh. Basically, this waste mixed fruit pulp is nothing but the leftover material obtained after extracting the juice from the whole fresh fruits (like oranges, pineapple, citrus limetta, sapota, watermelon, pomegranate, etc.). The percentage of different component of fruits is shown in Table 1. From Table 1, it can be concluded that on an average about 17.67% (4.71% to 28.06%) fruit pulp waste could be yielded from per kilogram of fruit.

Preparation of Sample

To make homogeneous slurry of collected mixed fruit pulp waste, it was blended in a mixture grinder. ASTM Standard (ASTM E1756-08(2015)) Standard Test Method for Determination of Total Solids in Biomass was followed to know the moisture content in the fruit pulp waste. For this purpose, it was kept in an oven at 105 ± 5°C for 4 hours. Later, dried mixed fruit pulp waste was ground with the help of a mixer grinder. To ensure the uniform size of ground powder, it was sieved using a 2 mm size sieve and stored in a desiccators for further experimentation [10].

Proximate Analysis of Dried Mixed Fruit Pulp Waste

Proximate analysis of powdery mixed fruit pulp waste was carried out using ASTM standard. Ash content (E-1755, 2015) and volatile matter (E-872, 1998) were determined using muffle furnace and difference of ash content and volatile matter from 100 on dry basis gives the fixed carbon (E-1755, 2001 and E-872, 1998). A bomb calorie meter was used to know the calorific value of dried and powdered sample.

Fourier Transform Infrared Analysis of Powdered Waste Mixed Fruit Pulp

Fourier transform infrared spectroscopy (FT-IR) of dried powder of waste mixed fruit pulp was recorded from an FT-IR spectrophotometer (model Tensor 27, Bruker). The powdered sample was scanned between the wavelengths of 4000 and 400 cm^{-1} at a resolution of 2 cm^{-1} in the transition mode. The generated FT-IR spectra of powdered waste fruit pulp sample were analyzed and compared to already published FT-IR spectra of lignin and cellulose to determine the key functionalities present in the sample [11].

Preparation of Chemicals for Hydrolysis

Sulfuric acid was selected as the hydrolysis chemical for hydrolysis of powdered waste mixed fruit pulp. Different concentrations of sulfuric acid were prepared (20%, 25%, 30%, 35%, 40%, and 45%), by mixing distilled water with 98% concentrated sulfuric acid. Concentration of acid was calculated as per the method suggested by Singh and Asha [11]. Mathematically it is written as

$$\mathrm{QCAR} = \frac{\mathrm{DAC} * \mathrm{DSQ}}{\mathrm{PA}}$$

Where, QCAR = quantity of concentrated acid required for the desired concentration, DAC = desired acid concentration in percentage, DSQ = desired solution's quantity in milliliters (mL), and PA = purity of acid.

Table 1. Weight of pulp extracted per kilogram of fruit.

Fruit, 1000 g	Juice, g	Fruit peeling, g	Pulp after juice extraction, g	Losses, g
Oranges	500	219	280.67	0.33
Citrus limetta	421	276	302.88	0.12
Pomegranate	725.3	198	76.68	0.02
Pineapple	639	311.81	47.094	2.069

Figure 1. Sample preparation.

Using the above formula, all the acid solutions were prepared (Figure 1).

Experiment Process

For the hydrolysis process, 50 g of dried and powdered fruit pulp was mixed with 125.51 mL of 25% concentrated H_2SO_4 and the solution was gradually mixed by manual stirring with the help of a glass rod (Figure 2). A dark brown homogenous paste was obtained by continuous mixing of this mixture. Later 125 mL of heated (100°C) distilled water was added to the homogeneous paste. This mixture was regularly stirred and heated at a temperature of 50°C for 30 minutes. A black liquor (Figure 3) was obtaines, which was allowed to cool to room temperature and filtered using a cotton filter cloth. By filtration, insoluble solids are separated in the form of the solid cake leaving with black liquor.

The black liquor was heated for 2 hours at 50°C in an oil bath. Later, this black liquor was distilled using fractional distillation (Figure 4), maintaining 78°C temperature, as the boiling point of ethanol is 78°C. The resulting vapours were condensed and distillate is obtained which might contain traces of water [12].

Figure 2. Dark brown homogenous paste.

Figure 3. Black liquor.

Figure 4. Distillation of black liquor.

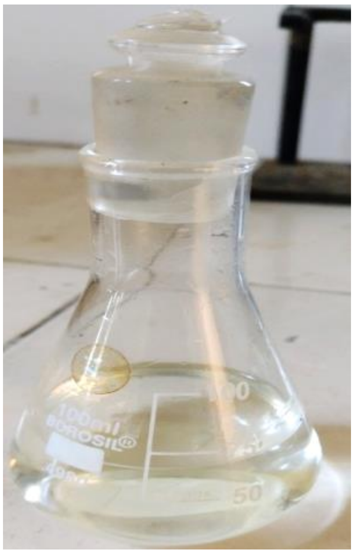

Figure 5. Bio-ethanol produced in the laboratory using mixed fruit pulp waste.

The amount of water present in the bio-ethanol after distillation was removed through dehydration, which was performed with anhydrous sodium sulphate at the ratio of 1:2 of distillate (Figure 5). The above process was repeated by treating the black homogeneous paste of 25% concentrated H_2SO_4 and 50 g of powdered mixed fruit waste pulp sample at different temperatures (60°C, 70°C, 80°C, and 90°C). Further, the whole experimental process was also repeated with 30%, 35%, 40%, and 45% concentrated H_2SO_4 solution at different temperatures (60°C, 70°C, 80°C, and 90°C).

RESULTS AND DISCUSSION

Proximate Analysis of Powdered Waste Mixed Fruit Pulp

Results of the proximate analysis of powdered waste mixed fruit pulp are presented in Table 2. A close look of Table 2 clearly indicates that mixed fruit pulp has less ash content and high energy content, which may make it suitable raw material for extraction of bio-ethanol.

Hydrolysis of Mixed Fruit Pulp

A total of 25 samples each weighing 50 g of powdered waste fruit pulp were taken for the study. The sample was hydrolyzed with different concentrations of H_2SO_4 at different temperatures for 2 hours. Detailed results are tabulated in Table 3 and also shown graphically (Figures 6 and 7).

A close look at Table 3 and Figures 6 and 7 indicates that at a temperature of 70°C with 35% (v/v) sulfuric acid concentration, the maximum yield of bio-ethanol (560 mL/kg of fresh mixed fruit pulp waste) could be obtained. It may be because the fibrous structure of the pulp might have broken at this particular temperature without dissolving the cellulosic material and subsequently the sugar. At the 35%

Table 2. Proximate analysis of powdered waste mixed fruit pulp.

Parameters	Values
Moisture content of fresh mixed fruit pulp waste, % wb	60.71
Moisture content of dried mixed fruit pulp waste, % db	2.5
Ash content, % db	2.7
Volatile matter, % db	78.01
Fixed carbon, % db	16.79
Calorific value, kCal/kg	23.54 MJ/kg

db, dry basis; wb, wet basis.

Table 3. Bio-ethanol yield at different H_2SO_4 acid concentrations and temperature.

Temperature, °C	% Concentration of H_2SO_4				
	25%	*30%*	*35%*	*40%*	*45%*
50	19	20	34	39	31
60	26	34	43	54	33
70	30	49	71	67	40
80	32.5	44	41	39	34
90	17	17.7	20	18	16

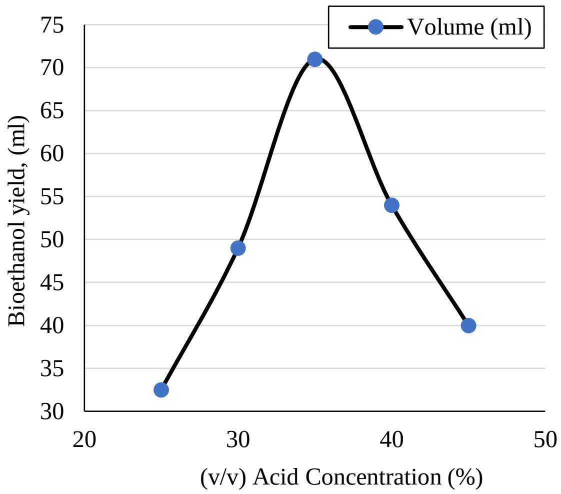

Figure 6. Effect of acid concentration on bio-ethanol yield.

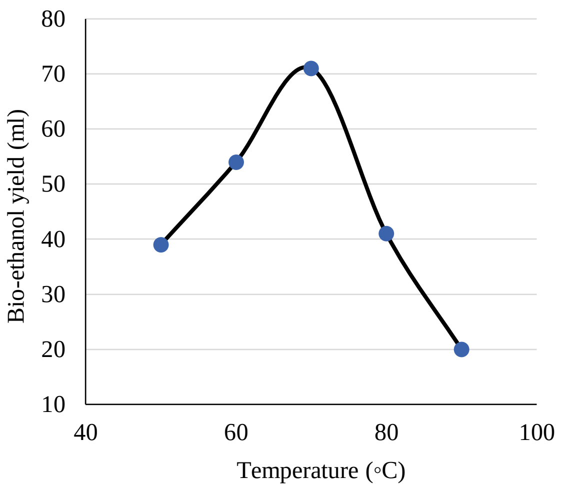

Figure 7. Effect of temperature on bio-ethanol yield.

Table 4. Properties of bio-ethanol produced from the waste mixed fruit pulp.

Properties	Bio-ethanol (Standard)	Bio-ethanol (Resultant)
Purity	100%	92%
Calorific value	27 MJ/kg	21.54 MJ/kg
Moisture content, % wb	-	43.714
Density	0.789 g/mL (at 25°C)	0.845 g/mL (at 25°C)
pH	6.8	6.7
Boiling point	78.4°C	78.8°C

of the concentration of acid, the cellulose and the sugar get dissolved and might have yielded into some other complex compounds of carbon. The temperatures below 70°C may not be sufficiently enough to break lignocelluloses structure of the fruit pulp and as a result of which not yielded into the optimum amount of bio-ethanol.

Physical and thermal properties of bio-ethanol obtained from waste mixed fruit pulp were analyzed as per the standard methods and the results are compared with standard bio-ethanol (Table 4). Results indicate that most of the properties of bio-ethanol obtained from waste mixed fruit pulp are at par with standard bio-ethanol except for energy content, which may be due to the presence of moisture in the resultant bio-ethanol.

FT-IR Analysis

To identify the unknown compounds and the composition of pre-treated powdered mixed fruit pulp waste, FT-IR spectroscopy was performed. Critical analysis of FT-IR spectra (Figure 8) indicates that waste fruit pulp is mainly composed of cellulose, hemicelluloses, and lignin with functional groups like aromatic ketones, and alcohols.

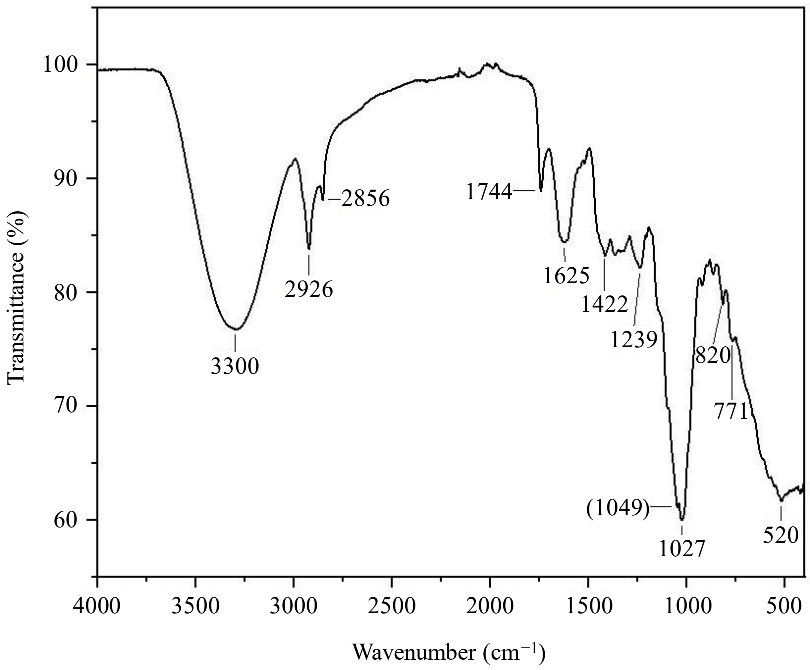

Figure 8. Fourier transform infrared (FT-IR) spectra of the powdered sample of waste mixed fruit pulp.

Table 5. Summary of Fourier transform infrared (FT-IR) spectra analysis.

Band number	Wave number, cm^{-1}	Assignments
1	3300	O-H stretching
2	2926	-CH stretching of -CH2 and -CH3
3	2856	Methylene C-H symmetrical Stretch
4	1744	C=O stretching of un-conjugated ketone, carbonyls, and Easter groups, C=O in xylan acetates (hemi-cellulose)
5	1625	Aryl-substituted C=C stretching
6	1422	Aromatic skeletal vibrations combined with C-H in plane deformation
7	1239	Syringyl ring breathing and C-O stretching out of lignin and xylan
8	1049	Primary alcohol C-O stretching in cellulose and hemi-cellulose
9	1027	Cyclo-hexane ring vibrations
10	820	C-O-O- stretch of ether or oxy compound
11	771	Aromatic C-H out-of-plane bend
12	520	Aliphatic iodo compounds, C-I stretch

A close look at Figure 8 indicates that there are three peaks in the single bond region at 3300, 2926, and 2856 cm^{-1} are available with mixed fruit pulp waste. The peak at 3300 cm^{-1} shows the presence of –OH vibration from alcohol and pectic acid group in the composition of biomass. The peak at 2926 cm^{-1} corresponds to the C–H asymmetrical stretching vibrations of aliphatic structures, and the peak at 2856 cm^{-1} represents methylene C–H symmetrical stretch. In the double bond region peaks identified at 1744 cm^{-1} represent the presence of hemicelluloses structure in sample composition and the peak at 1625 cm^{-1} represents aryl-substituted C=C stretching. The vibrations of aliphatic chains of basic cellulosic materials are seen at 1422 cm^{-1}. C–O stretching has been seen due to peaks at 1239, 1049, and 1027 cm^{-1}. Peak at 820 cm^{-1} signifies a small C–O–O stretch of ether or oxy compound, a peak at 771 cm^{-1} represents an aromatic C–H band, and a peak at 520 cm^{-1} indicates a small C–I stretch. The summary of FTIR spectra analysis is presented in Table 5.

CONCLUSION

1. Waste mixed fruit pulp has been proven a potential feedstock for the production of bio-ethanol. Apart from that, it is locally available in any part of India.
2. H_2SO_4 concentration of 35% at 70°C temperature gives optimum conditions for maximum recovery of bio-ethanol.
3. Properties of waste fruit pulp bio-ethanol are at par with pure ethanol.
4. At optimum conditions (35% H_2SO_4 concentration and 70°C temperature) about 560 mL bio-ethanol could be obtained from 1 kg of fresh waste mixed fruit pulp.

Declaration of Interest Statement

It is declared that authors do not have any known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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