

Renewable and sustainable fuel production from woody biomass

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Bioethanol has been produced from woody biomass (Neem wood saw dust) and the work has been extended to check the feasibility of Honge oil methyl ester (HOME)-bioethanol blends. Based on the experimental results, 11.8 g/L glucose is obtained with H₂SO₄ acid hydrolysis at 120°C reaction temperature, 2% H₂SO₄ concentration and 120 min reaction time for pretreated biomass. The results for batch fermentation show that pH 4.5, temperature 30°C and incubation period of 72 h are found to be favourable for producing maximum bioethanol. Based on the results obtained on HOME-Bioethanol blends, bioethanol from woody biomass has been found to be feasible to use in blend form.

Keywords: Bio-ethanol, Woody biomass, Honge oil methyl ester, *Sacchromyces cerevasiae*, Honge oil methyl ester-bioethanol blends.

During last two decades, due to rapid industrialization and economic development, pollution, energy crisis and global warming have emerged as most important challenges globally. These problems have led to an increased awareness regarding necessity to adopt sustainable solution for energy alternatives and so many countries are using renewable and alternative fuels. Biofuels such as biodiesel derived from vegetable oil and bioethanol from biomass are the most promising alternative fuels for engine application, because these are renewable, environment-friendly, clean burning, non-toxic and biodegradable. Biofuels also have no sulphur and aromatics so they have favorable heating value and cetane number. But it has been found that neat vegetable oil poses some problems when it is used directly as diesel substitute¹⁻³. These problems can be solved by blending the vegetable oil/biodiesel with bioethanol. Bioethanol production and utilization in a diesel engine have been reported. They were found acceptable power output with increased smoke, HC and CO and decreased NOx emission levels¹. The production of ethanol from lignocellulosic biomass has been reported⁴. Lignocellulosic biomass contains 35-50% cellulose, 25-30% hemicelluloses and 20-25% lignin⁵. Producing ethanol from corn or sugar cane (or other sugar/starch crops) is less technically challenging than producing ethanol from

lignocellulose in woody materials^{6,7-12,13}. These sources have widespread abundance and the cost of their procurement is relatively cheap.

However, bioethanol production from woody biomass is less investigated as this area needs detailed study on the production of bioethanol. Production of ethanol from woody biomass was investigated during the year 1819¹⁴⁻¹⁶. Further it was not investigated in detail. It may be due to unavailability of resources and technology. They possess several characteristics that could propel them to the forefront of the renewable fuels industry and provide sustainable solution for energy crisis, air pollution and climate. Use of biofuels for engine applications, marginally decreased performance with reduced emission levels has been reported^{1,17}.

Wood sawdust is a lignocellulosic biomass which is a byproduct of sawmill that is available at low cost throughout the year. It is produced in enormous quantities by sawmills and the economical disposal of them is a serious problem to the wood based industries. Sawdust is commonly used as fuel, bedding material in poultry and livestock structures and also for the production of fiberboards and paper pulp¹⁸⁻²⁰. Hence, woody biomass are among the most promising and renewable non-food-crop-based alternative.

Many investigators reported that acid hydrolysis and enzymatic hydrolysis as the conversion method

from woody biomass to bioethanol^{1,21,22}. In general, to produce bioethanol, first, woody biomass was pretreated then hydrolyzed using acid followed by fermentation process in presence of microorganisms. A major advantage to cellulosic bioethanol is that, it has lower carbon and sulfur emissions than fossil fuel.

Bioethanol production from woody biomass requires pretreatment of biomass. Studies on the different pretreatment methods revealed that different pretreatment methods produce different effects on the biomass in terms of its structure and composition. Physico-chemical method such as steam explosion method is recognized as one of the most cost-effective for hardwood and agricultural residues, but is less efficient for softwood²². The main advantages of steam explosion is that, it is more suitable for hard wood, requires little biomass-size conditioning and process generate less hazardous process chemicals, and has potential for lowering environmental impact, and lower capital investment^{22,23}. But, it needs high pressure steam. The major differences between woody and non-woody (agricultural) biomass are their physical properties and chemical compositions. The main objective of the present study includes bioethanol production from woody biomass using steam explosion pretreatment method and various process variables affecting the bioethanol production.

Experimental Section

The raw material was oven dried at 105°C until constant weight and stored in a sealed plastic bag at ambient temperature for chemical analysis. Cellulose, hemicellulose and lignin of Honge, Neem and Babul wood were measured according to the procedures

reported in the literature²⁴. In this present work, for bioethanol production, a suitable wood saw dust (Neem wood) was selected based on the extractives, availability and composition and it was obtained from local saw mill.

Pretreatment

In the present work, bioethanol was produced from woody (Neem) biomass using hydrothermal explosion pretreatment followed by acid hydrolysis, fermentation and separation. Figure 1 shows schematic diagram of bioethanol production from babul woody biomass.

The biomass (saw dust) was collected, dried and sealed in an air tight container. Pretreatment for the biomass was performed in a laboratory-scale steam explosion unit which is provided with temperature and pressure indicators. Known quantity (50 g) of feedstock was loaded in to the unit and no chemicals were used in the explosion unit. In the process of steam explosion, biomass is treated with hot steam (more than 150°C at pressure of 5 bar) under pressure followed by an explosive decompression of the biomass that results in a rupture of the rigid biomass fibers. The major physicochemical changes of biomass during the steam-explosion are attributed to the hemicelluloses removal and lignin transformation²⁵. During the experimentation, the steam explosion unit was kept sealed and steam was admitted and maintained at a temperature more than 150°C. The biomass is retained at this condition for more than ten minutes and suddenly depressurized to atmospheric pressure. The pretreated material was collected, filtered, washed with 200 mL of hot

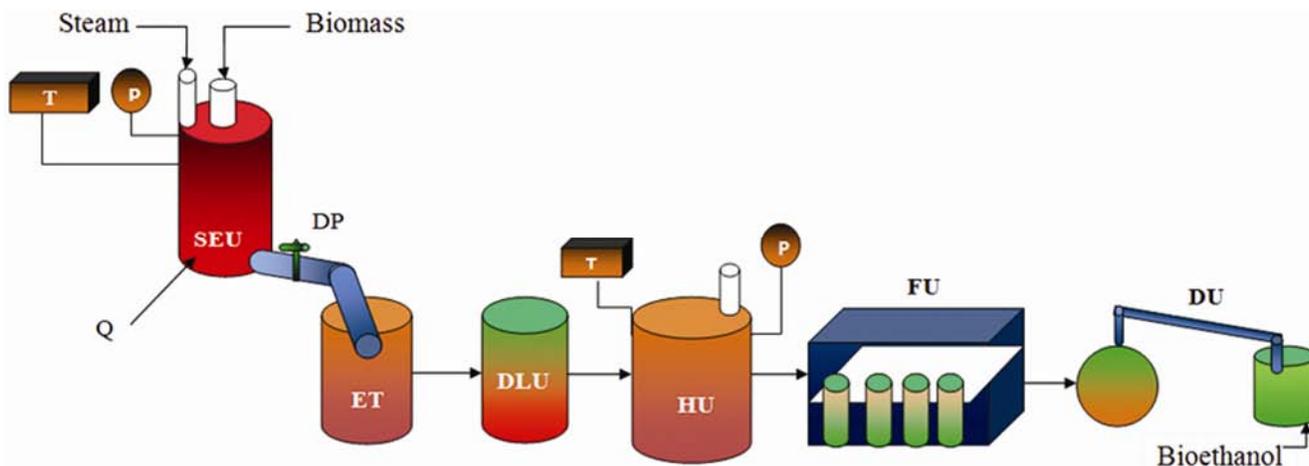


Fig. 1— Block diagram of bioethanol production, T- Temperature sensor, P – Pressure sensor, SEU – Steam explosion unit, ET- Explosive tank, DLU- Delignification unit, HU-Hydrolysis unit, FU – Fermentation unit (Rotary incubator), DU–Distillation unit.

water and dried. The objective of a steam explosion pretreatment is to make the cellulose better accessible for hydrolysis. Increase in temperature up to a certain level can effectively release hemicellulosic sugars²⁶.

Hydrolysis

Solids are then delignified using 10% NaOH for 2h, followed by hydrolysis with different acid concentrations (in steps 0.5%) and reaction time (60, 90 and 120 min) at 120°C. Hydrolysis (saccharification) process breaks down the hydrogen bonds present in the cellulose fractions into fermentable sugar components. The product of hydrolysis was then filtered using vacuum filter. Liquid filtrate obtained was neutralized with calcium carbonate and then treated with activated charcoal (1:40 w/v), for removal of any degraded compounds (furfurals) formed. Later glucose is quantitatively estimated using DNS method.

Fermentation

For the fermentation of sugar, a suitable media was prepared by adding 7.5 g ammonium sulphate ((NH₄)₂SO₄), 5 g potassium hydrogen phosphate (KHPO₄), 1.5 g magnesium sulphate (MgSO₄) and 2 g calcium chloride (CaCl₂·2H₂O) to 1000 mL of distilled water and dissolving completely. Media prepared was then added to the sugar solution and 5% yeast (*Saccharomyces cerevisiae*) was inoculated to the hydrolyzate and kept for fermentation at different temperatures for 3 days in the rotary incubator at a speed of 200 rpm. Also the variation of bioethanol yield with different pH was studied during fermentation. After fermentation, the broth was centrifuged to separate the solid biomass; the supernatant obtained was later distilled at 78°C to separate bioethanol.

Results and Discussions

The characterization of fuels, factors affecting the bioethanol production and feasibility tests has been reported in the subsequent paragraph.

Characterization of the fuels used

Neem has medicinal use, hence in the present work, waste saw dust from Neem wood (saw mill waste) was chosen for the study as it is abundantly available locally compared to other woods tested. Bioethanol and its blends properties and composition of different wood are given in Tables 1 and 2.

Acid hydrolysis

Acid hydrolysis for pretreated biomass was conducted with different reaction conditions, like

reaction time, acid concentration and temperature, to determine the optimum parameters for maximum glucose yield.

Figure 2 shows the glucose conversion with different acid concentrations. Batch experiments were conducted using 2M H₂SO₄ as the reaction catalyst at maximum reaction temperature of 120°C and hydrolysis of biomass was conducted at various concentrations of acid ranging from 0.5 to 2.5% in steps of 0.5% and at reaction time of 60, 90 and 120 min respectively. Results showed that, high acid concentration enhances rate of reaction while improving the sugar concentration as acid catalyze the hydrolysis of biomass. Since catalyst activity was proportional to hydrogen ion [H⁺] concentration, formation of more hydrogen ions in the solution resulted in higher glucose yield during cellulosic hydrolysis process. It could be attributed to increased reaction rate due to higher breaking of glycosidic bonds. Hence it causes high conversion of cellulose fraction into glucose.

From Fig. 2, it is observed that, degradation of glucose leads to furfurals under sever conditions i.e. with 2.5% H₂SO₄ acid concentration and 90 min reaction time. However, at constant reaction temperature of 120°C, 11.8 mg/mL of glucose were resulted with 2% H₂SO₄ respectively, with 120 min reaction time (Fig. 2). Results indicated lower yield with higher concentration of H₂SO₄ and longer reaction time. It may be due to deformation of sugar to hydroxyl methyl furfurals. Based on the results obtained it could be concluded that, the acid concentration was found to be more dominating effect than reaction time during hydrolysis. Higher yield may also be due to higher temperature and it provides greater energy to break linkages of fibers in a biomass.

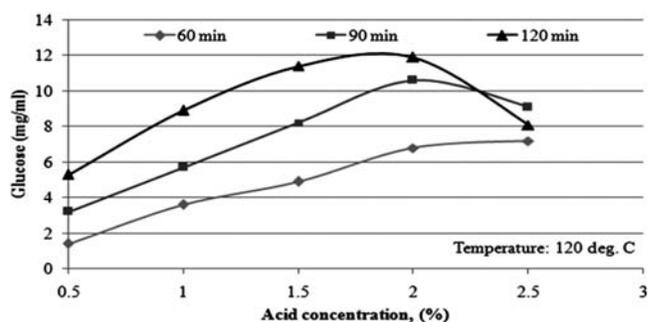


Fig. 2—Glucose yield at varied acid concentration for different time interval.

Table 1—Property of Diesel, HOME and its blends with bioethanol

Property	Diesel	Ethanol	HOME	BE 5	BE 10	BE15	ASTM test No
Density(kg/m ³)	840	790	870	880	870	862	D4052
Kinematic Viscosity (cst) @ 40°C	3.5	1.0016	5.84	5.0	4.8	4.4	D445
Flash point (°C)	56	12	170	152	146	140	D93
Calorific value (kJ/kg)	43,000	21,200	36100	35958	35896	35834	----

Table 2—Composition of Wood

Wood	Lignin wt%	Cellulose wt%	Hemicellulose wt%
Ordinary or Babul	25.1	49.6	18.4
Neem	26.7	47.8	16.1
Honge	25.4	42.24	15.6

Fermentation

Figure 3 shows that *pH* of medium during fermentation is an important parameter affecting the growth and product formation. Results reveal that bioethanol yield is higher for a reaction with *pH* of 4.5. For the fermentation process, fermentation time was fixed based on the published literature²⁷. It is observed that yield was reduced with increase in *pH* of media. It could be attributed to neutral or basic *pH* of media and greatly inhibit the overall yeast health. In the present work, Baker's yeast was used as it has an internal *pH* of about 5.0 which favors reproduction and growth in a slightly acidic environment. Finally, it is observed that lower *pH* levels ensure that the yeast function well under minimal internal stress. Optimum value in terms of *pH* was found to be 4.5 at 72 h for maximum sugar conversion of 52%.

Figure 4 represents the effect of temperature on bioethanol yield during fermentation stage. Optimum temperature for maximum yield of 52% was found to be 30°C. It could be due to the fact that the yeast was under minimal stress and lower inhabitation from bioethanol obtained. The yeast cells at 30°C are structurally sound and are capable of healthy and efficient reproduction. It is observed that beyond 30°C, the yield was lowered because of lower reaction kinetics. Also, it may be due to high temperature resulting in considerably higher thermal stress on the yeast as it reproduces and slower reproduction rate of yeast to consume the available glucose substrate. Hence, it is found that lower temperature favors bioethanol fermentation.

Feasibility of Biodiesel-Bioethanol (BE) blends

The feasibility of produced bioethanol from saw dust was checked for engine application by measuring

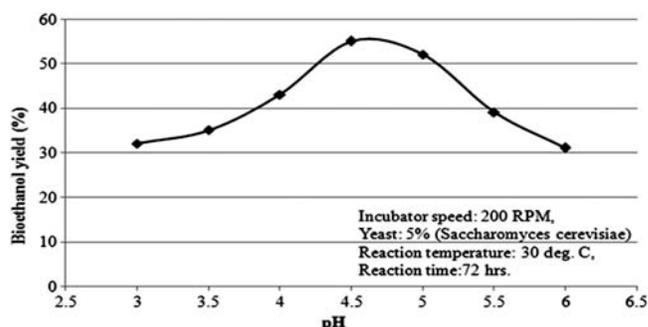


Fig. 3—Variation of bioethanol yield with pH.

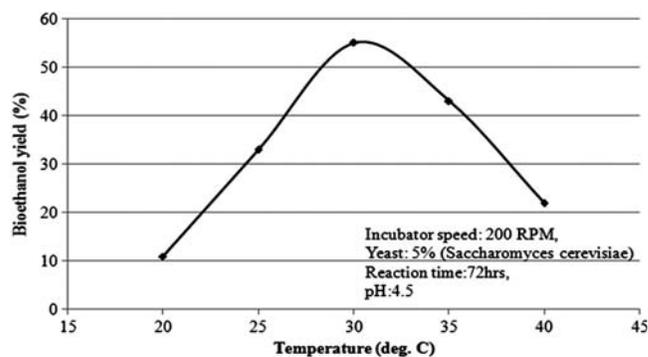


Fig. 4—Variation of bioethanol with temperature

various biodiesel-bioethanol (BE) blend properties. For the present work, different blends such as HOME+5% bioethanol (BE5), HOME+10% bioethanol (BE10), HOME+15% bioethanol (BE15) and HOME+20% bioethanol (BE20) were used.

Kinematic viscosity

Raw non-vegetable oil (Honge oil) has significantly higher viscosity at normal temperature and slight compressibility than edible vegetable oils. This feature makes the oil difficult during extraction and injection. In the present work, the kinematic viscosity (KV) was measured by Redwood viscometer. The indication of high viscosity affects the fluidity of the oil and it leads to poor atomization. Hence, it leads to incomplete combustion. To reduce high viscosity of raw oil, the oil must be converted in to their respective biodiesels. However, to enhance diesel engine performance, it is necessary to blend the

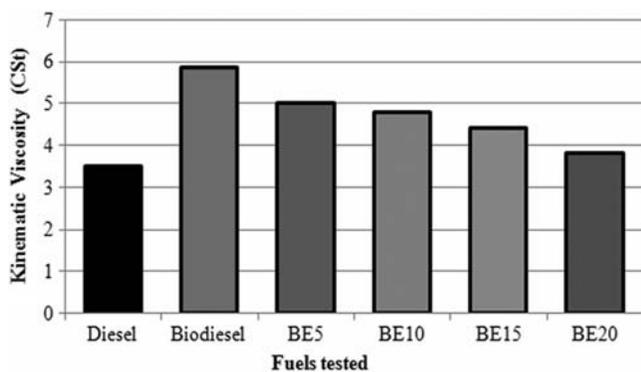


Fig. 5—Variation of KV with bioethanol proportion

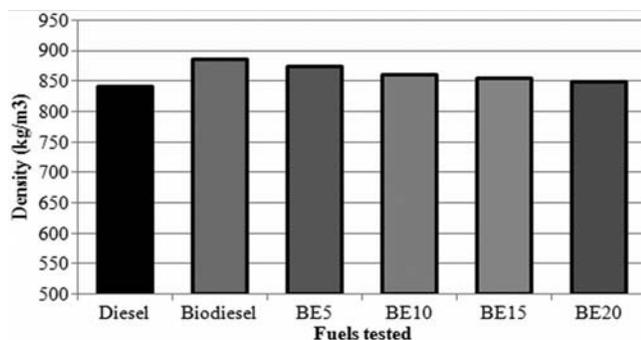


Fig. 6—Variation of density with bioethanol proportion

biodiesel with bioethanol. Therefore it is better to use bioethanol for blending with biodiesel. This could make a blend more suitable for diesel engine application. Figure 5 shows the effect of bioethanol on the KV. After blending, the values of KV were decreased by 18, 12 and 4% compared to BE5 respectively at 40°C. Blending brings the viscosity of biodiesel near to that of diesel fuel. It is observed that, the higher viscosity for biodiesel; this is because of higher molecular attraction of the long chains of its triglyceride molecules. This factor increases the chances of carbon deposition in the combustion chamber.

Density

Figure 6 show that the density of oil tested decreases with increase in bioethanol content. The density of biodiesels was found to be higher than neat diesel fuel. This may be due to presence of triglycerides in oil. The density of biodiesels can be reduced by blending with bioethanol. By adding 5-20% ethanol (volume basis) in steps of 5%, blending reduces a specific gravity of about 1-1.6%. It was observed that 2.7, 2.0 and 1.12% decreased density for BE10, BE15 and BE20 compared to that of BE5 respectively at 40°C.

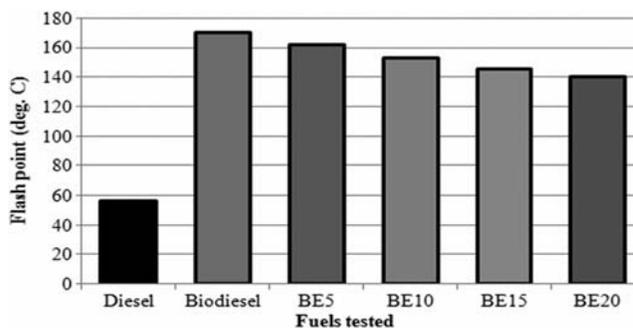


Fig. 7—Variation of flash point with bioethanol proportion

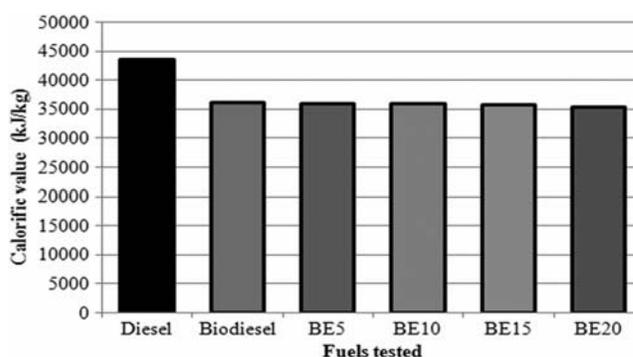


Fig. 8—Variation of calorific value with bioethanol proportion

Flash point

The flash point of biodiesel was found to be higher than diesel (Fig. 7). From the results, it is observed that, the flash point of BE10, BE15 and BE20 were decreased by 11.2, 7.9 and 3.9% compared to BE5 respectively.

Calorific value

The variation of calorific value with various HOME-bioethanol blends is shown in Fig. 8. It was found to be approximately 10-11% lesser than diesel fuel. This is because of presence of moisture (oxygen) in a biodiesel. The calorific value of BE5, BE10, BE15 and BE20 were found to be 35958, 35896, 35634 and 35284 kJ/kg respectively. It is observed that, the flash point of BE10, BE15 and BE20 were decreased by 1.9, 0.9 and 0.2% compared to BE5 respectively.

Conclusion

The following conclusions have been made from the study. Optimal conditions for H₂SO₄ acid hydrolysis are found to be 120°C reaction temperature, 2% H₂SO₄ concentration and 120 min reaction time for glucose yield of 11.8 mg/mL. For bioethanol production, the optimum values

for fermentation process are found to be 30°C temperature, pH of 4.5 and 72 h reaction time for maximum yield of 52%. In a fermentation process with yeast of *Saccharomyces cerevisiae*, beyond 30°C temperature the yield of bioethanol is lowered. Based on the results obtained on HOME-Bioethanol blends, bioethanol from woody biomass is found to be feasible to use in blend form. The properties of bioethanol are found very close to ethanol derived from molasses. Use biodiesel from non-edible vegetable oil and bioethanol from waste (neem saw dust) avoids the conflict between food and energy security.

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