Characterization of biodiesel production from sugar cane filter mud oil – Wonji sugar factories in Ethiopia

Pedro Dionisio Remedios Castañeiras, Abowerk Wagank, Hailemariam Nigus Hailu

Abstract

Significant improvements to Ethiopia's trade balance are needed to required economic development. One main issue is that around 65% of Ethiopia export earnings are needed to pay for the import of petroleum products. Biodiesel can be produced from plenty of oil containing feedstock and is an alternative renewable fuel. This research was about the production of biodiesel from sugarcane filter mud (cake) oil that it is non edible. During this work the biodiesel was produced at standard material and at standard condition. The experiments were conducted using filter mud, and cold n-hexane as extracting solvent at temperature less than 18 ºC. The time used for extraction of filter mud oil was 2, 4, 6, and 8hours and a filter mud particle size between 1 to 2mm. the maximum yield 4g of filter mud oil was obtained, at the extraction time of 6hour. After the extraction of filter mud oil the next step was the transesterification of this oil to obtain biodiesel. The influence of methanol to filter mud oil, catalyst to filter mud oil weight ratio, reaction temperature, the rate of mixing and the reaction time was examined. The higher heating value and Cetane number of biodiesel obtained from filter mud oil were found to be 44.18MJ/kg and 81.5 respectively.

Key words: Vegetable oil; diesel; biodiesel, Sugar cane filter cake oil.

1. INTRODUCTION

Biodiesel is the name for a variety of ester based fuels (fatty ester) generally defined as mono alkyl ester made from renewable biological resources such as vegetable oils (both edible and non-edible), recycled waste vegetable oil and animal fats. This renewable source is as efficient as petroleum diesel in powering unmodified diesel engine. Today diesel engines require a clean burning, stable fuel operating under a variety of conditions. Using biodiesel not only helps maintaining our environment, it also helps in keeping the people around us healthy. Biodiesel is miscible with petro diesel in all ratios. In many countries, this has led to the use of blends of biodiesel with petro diesel instead of neat biodiesel [7].

Biodiesel derived from biological resources is a renewable fuel, which has drawn more and more attention recently. A fatty acid methyl ester is the chemical composition of biodiesel. Transesterification is widely used for the transformation of triglyceride into fatty acid methyl ester [9]. The manufacturing process is based on the transesterification of triglycerides by alcohols to fatty esters, with glycerol as a byproduct [10]. In this way, highly viscous triglycerides are converted in to long chain monoesters presenting much lower viscosity and better combustion properties. Homogeneous or heterogeneous catalysis are used to enhance the reaction rate. Raw materials are vegetable oils, preferably non-edible, but also different wastes, such as used frying oils or animal fats [15].

There are different types of feed stocks that are used for the production of biodiesel. These includes linseed oil, palm seed oil ,waste cooked vegetable oil, sun flower seed oil, cotton seed oil, Jatropha seed oil, vernonia galamensis seed oil and animal fats. Oil seed plants are used for the production of biodiesel through the process called transesterification reaction which is a process by which alcohol reacts with vegetable oil in the presence of catalyst. Triglycerides are major components of vegetable oils and animal fats. Chemically, triglycerides are esters of fatty acids with glycerol. Fatty acid ethyl esters are mostly involved because ethanol is the cheapest alcohol, but other alcohols, namely methanol, may be employed as well. In this way, highly viscous triglycerides are converted in long chain mono esters presenting much lower viscosity and better combustion properties to enhance the burning. Homogeneous or heterogeneous catalysis are used to enhance the reaction rate. Sugar cane mud (Saccharumofficinarum, L.) is a residue of the Sugar Cane Industry obtained by vacuum filtration of the grounds that flocculate when sugar cane juice will be clarified, with the objective of to eliminate non-soluble impurities contained on it. Mud composition is very variable, but in general it has a high content of soil, cane trash, saccharides and sugar cane wax.

Sugar cane wax is found near the sugar cane stem nodules, in the cuticle, its main components are high molecular weight esters, polyesters, fatty acids, dicarboxylic acids, aldehydes, ketones, alcohols and hydrocarbons [2]. During the sugar cane grinding process, most of the sugar cane wax is present in the juice; the former will be separated in the vacuum filtration process of the juice, forming part of the mud. The sugarcane industry has several co-products of immense potential. The co products include press mud (filter cake), molasses and spent wash. Press mud is a soft, spongy, amorphous and dark brown material containing sugar, fiber and coagulated colloids including cane wax, albuminoids, inorganic salts and soil particles. It consists of 80% water and 0.9 - 1.5 % sugar, organic matter, nitrogen, phosphorus, potassium, calcium, sulfur, coagulated colloids and other materials in varying amounts. Press mud like other organic materials affects the physical, chemical and biological properties of soil. However, due to its bulky nature and wax content it causes some problems.

Pedro Dionisio Remedios Castañeiras, PhD
Federal Technical Vocational Education & Training Institute (TVETI), Addis Ababa, Ethiopia. pdremedio63@gmail.com , Sub-city Suumit, Addis Ababa, Ethiopia

Abowerk Wagank, Bsc
Jimma Institute of Technology, Jimma University, Ethiopia. demeke@gmail.com , Kebele 05 , Jimma , Ethiopia

Hailemariam Nigus Hailu, MsC
Federal Technical Vocational Education & Training Institute (TVETI), Addis Ababa, Ethiopia. hailuqua@gmail.com , Sub-city Yeka, Addis Ababa, Ethiopia

2. MATERIALS AND METHODS

2.1 Raw Material

Materials

Materials and apparatus used in the production of the biodiesel are as follows: thermometer, retort stand, pipette, measuring cylinder, separating funnel, oven, water bath, hydrometer, conical flask, digital weighing balance, stop watch, hot plate, distilled water, methanol, n-hexane and sugar cane filter mud.

2.1.1 Raw material preparation

Filter mud was obtained from wonji sugar factories. Impurities like dusts and metals were removed by hand. Filter mud were crushed by hands with 1.0 mm to 2.0 mm sieve size in chemistry laboratory; then the sample was ready for oil extraction.

Figure 3.1(a). Filter mud collections from wonji sugar factory, Ethiopia

Samples analysis

Determination moisture (specific humidity) content of filter mud sample

It was used 50g, 100g, 150g and 200g of the cleaned sample was weighed and dried in an oven at 105°C and the weight was measured every 2hrs. The procedure was repeated until a constant weight was obtained.

The percentage moisture in the filter cake was calculated using the following Equation:

$$\text{Moisture \%} = \frac{W1-W2}{W1} \times 100\% \quad (1)$$

Where: W1 = original weight of sample before drying; W2 = Weight of sample after drying

Analysis of the filter mud and size reduction

The moisture was removed by placing the sample in an oven at 65°C for 4 hours. The dried filter mud was crushed in manually by hands with sieve size 1 to 2mm. The sample was sieved using hand shake with set of sieves sizes arranged in descending order 2mm, 1.75mm, 1.50mm, 1.25mm, and 1mm.

2.1.2 Oil extraction

Leaching extraction (Solvent extraction) [24]

In the research Reinosa [9], is obtained that the sugar cane filter cake oil has five time more antioxidant content (6%) than other vegetable oil like soy bean (1.5%), olive (0.5%) , sun flour (2%) approximately. There are presents, in sugar cane filter cake oil, some of important fatty acid to use oil as lubricant such as oleic and linoleic it was obtained. It is demonstrated that the sugar cane filter cake oil have good properties to production lubricant and good protect metals against corrosion.

The oil used during the research was supplied by the chemistry laboratory of Jimma University, where carried out the extraction process by leaching to using hexane as solvent, from plantations of sugar cane of Wonji Sugar Factory of Adama, Oromia Region of Ethiopia.

Solvent extraction is used for oil production from filter cake, because solvent extraction has higher oil yield when compared to other like mechanical extraction method for low content of oil. Experimental work was conducted using cold hexane solvent for extraction of oil, because hexane has high extraction ability when compare to other solvents and another component like wax and resin inside the sugar cane filter cake are not soluble or has lowest solubility in cold hexane [5, 18, 20].

Samples preparation

The ratio of solid to solvent that was used in this experiment (Figure 2 a) was 1:5, thus 500 ml of solvent was added to the beaker containing 100 g of filter cake powder. A stirrer in the bottom beaker was used to mix up the powder with the solvent thoroughly. During this process, the mixture was kept in cold weather the beaker in to the cold water with ice to control the temperature less than 20 ^{o}C. The solvent-oil mixture in the beaker was collected at the end of the extraction process.

Organic solvents for oil extraction Hexane (99% of purity) were used as organic solvents to leach out oils from the insoluble solid structure of the filter cake. During the selection of organic solvents, the solid to solvent ratio was maintained as 1:5 for the oil extraction. Temperature and mixing time were kept constant at less than 20^{o}C and 3 hours respectively, according to the suggestion parameters reported by Martínez [8], amount of 500 ml of cold hexane (less than 20 ^{0}C) was put into 1000ml container. 100g of the sample was placed in the beaker and was mixed with the dissolvent. This is continued for 3 hours. The experiment was repeated by placing the same amount of the sample again by varying particle sizes (1mm- 1.4mm, 1.4mm- 1.8mm, 1.8mm-2mm) respectively. The solvent was separated and recovery to be used again by rotary vacuum evaporator (figure 3), the weight of oil extracted was determined.

Fig. 2 Extraction of oil from filter cake by using dissolvent extraction method a) Leaching extraction Cold Solvent & Sugar cane filter cake oil b) Sugar cane filter cake oil extracted

Fig. 3 Rotary vacuum evaporator

2.1.3. Characterization of the extracted sugarcane filter cake oil

The physical properties of the extracted sugar cane filter cake oil such as specific gravity at 25℃, density, dynamic viscosity, kinematic viscosity, PH value was analyzed using standard procedures and chemical properties such as acid value, saponification value and iodine value also determined.

The oil mass extracted (figure 2 b) from Sugar cane filter cake oil shown table 1 carried out an experiment in the chemistry laboratory of Jimma University in Ethiopia, from 100g of filter cake processed.

Tabla.1 Mass extracted oil from Sugar cane filter cake.

Samples	Oil (g)
1	4.5
2	4.3
3	4.0
4	5.2
5	4.0
6	5.1

The extraction rate as it can see, obtained from the table 1, was around 4.5% of oil in this research; the extraction carried out for filter cake in Ethiopia are similarly to the result obtained in other country like Cuba, R.M.Torres [5], in Cuba; if it can be considered the sugar cane filter cake oil density around 896.1 kg/m^3, similar too with reported value [3, 5, 24].

It was reported in the research P.D.R.Castaneiras [4] , R.M.Torres [5], O.R.Espinosa [6] some properties of sugar cane filter cake oil that have not significant difference with the values obtained in this research.

Table 2 Some properties of the sugar cane filter cake oil

Properties	Magnitude
Acidity index (mg KOH / g oil)	23
Saponification index (mg KOH / g oil)	145
Iodine content (g iodine / 100 g oil)	83
Content of unsaponifiable matter (%)	20
Density, (kg / m 3) ASTM D 1298	896.1
Kinematic viscosity at 40 oC, cSt ASTM D 445	75*
Kinematic viscosity at 100 ° C, cSt ASTM D 445	14*
Viscosity index (VI)*ASTM 2270	197

*Viscosity of oil with 0.5% of content of sugar cane filter cake wax [7]

2.2 Transesterification and Production of biodiesel from sugar cane filter mud oil [13,14,15, 16, 17, 18, 19, 20, 21, 22, 23]

The filtered filter mud oil was heated up to a temperature of 60^{o}C in a heater to melt coagulated oil. The heated oil of 10ml was poured in to the conical flask containing catalyst – alcohol solution, and this moment was taken as the starting time of the reaction. The reaction mixture was then shaken by using shaker at a speed of 500 to 600rpm. The yield and characteristics of biodiesel was depending on the type of oil used due to variation in the fatty acid composition and other characteristics of oil. Taking in to this consideration aspect, the filter mud oils from inedible sources have been taken as a raw material for the preparation of biodiesel using NaOH catalyst and methanol alcohol.

The transesterification was carried out at a reflux of methanol, a stirrer, condenser, a 250ml capacity glass reactor, and thermostat. The effect of process variables (reaction temperature, methanol to filter mud oil ratio, and catalyst weight and rpm) was investigated by response surface methodology. Both main and interaction effects of the variables towards FAME yield was thoroughly studied as shown on the table (4.8) below.

Table 4.8, Experimental data

Std order	Run order	Reaction temperature (oC)	Methanol to filter mud ratio	Catalyst to filter mud oil weight(gr am)	Revolution per minute(rad)	Actual biodiesel yield	Predict e d biodiesel yield	Residuals
20	1	60	7.5	5.8	600	3.95	3.99	-0.045
23	5	60	6.75	5.8	500	3.95	3.96	3.801E-003
3	3	55	7.5	4	500	3.68	3.68	-1.775E-003
19	4	60	6	5.8	600	3.92	3.86	0.060
12	5	65	7.5	4	700	3.52	3.51	5.892E-003
7	6	55	7.5	7.6	500	3.88	3.89	-9.719E-003
17	7	55	6.75	5.8	600	3.91	3.84	0.070
29	8	60	6.75	5.8	600	3.96	3.97	-8.088E-003
9	9	55	6	4	700	3.61	3.62	-0.013
8	10	65	7.5	7.6	500	3.78	3.76	0.018
4	11	65	7.5	4	500	3.58	3.56	0.021
14	12	65	6	7.6	700	3.46	3.45	7.336E-003
30	13	60	6.75	5.8	600	3.97	3.97	-6.088E-003
26	14	60	6.75	5.8	600	3.96	3.97	-9.088E-003
22	15	60	6.75	7.6	600	3.96	3.99	-0.030
6	16	65	6	7.6	500	3.47	3.46	0.013
15	17	55	7.5	7.6	700	3.86	3.84	0.025
27	18	60	6.75	5.8	600	3.96	3.97	-7.088E-003
11	19	55	7.5	4	700	3.57	3.59	-0.022
28	20	60	6.75	5.8	600	3.96	3.97	-0.012
21	21	60	6.75	4	600	3.92	3.87	-0.046
13	22	55	6	7.6	700	3.63	3.66	-0.031
5	23	55	6	7.6	500	3.71	3.71	-3.304E-004
2	24	65	6	4	500	3.44	3.46	-0.019
10	25	65	6	4	700	3.42	3.42	2.807E-004
24	26	60	6.75	5.8	700	3.93	3.91	0.019
1	27	55	6	4	500	3.69	3.71	-0.017
16	28	65	7.5	7.6	700	3.76	3.75	7.947E-003
18	*29*	*65*	*6.75*	*5.8*	*600*	*3.62*	*3.67*	*-0.054*
25	*30*	*60*	*6.75*	*5.8*	*600*	*3.97*	*3.97*	*-4.088E-003*

3. RESULT & DISCUSSION

Effect of process variables on transesterification reaction

Temperature, methanol to filter mud oil ratio, and catalyst weight was considered as input process variables. Analysis of variance on table shown below that was mains and interaction effect was significant in this model and variation of those process variables affects the product greatly.

Table 4.9 ANOVA for response surface Quadratic model.

Source	Sum square	Df	Mean square	F value	P value	Prob> F
Model	1.06	14	0.075	52.89	<0.0001	Significant
A-reaction temperature	0.12	1	0.12	86.43	<0.0001	Significant
B-methanol to filter mud oil ratio	0.084	1	0.084	59.09	<0.0001	Significant
C-catalyst to filter mud oil ratio	0.065	1	0.065	45.74	<0.0001	Significant
D-RPM	9.988E-003	1	9.988E-003	7	0.0184	Significant
AB	0.016	1	0.016	10.95	0.0048	Significant
AC	2.500E-005	1	2.500E-005	0.018	0.8965	N.S
AD	2.025E-003	1	2.025-003	1.42	0.2521	N.S
BC	0.042	1	0.042	29.45	<0.0001	Significant
BD	2.500E-005	1	2.500-005	0.018	0.8965	N.S
CD	1.225E-003	1	1.225E-003	0.86	0.3689	N.S
A^2	0.12	1	0.12	83.80	<0.0001	Significant
B^2	5.930E-003	1	5.930E-003	4.16	0.0595	Significant
C^2	4.113E-003	1	4.113E-003	2.88	0.1102	N.S
D2	4.113E-003	1	4.113-003	2.88	0.1102	N.S
Residual	0.021	15	0.021			
Lack of fit	0.021	10	0.021	286.19	<0.0001	Significant

Regression model equation

The model equation that correlates the response yield of filter mud oil to biodiesel to the transesterification process variables in terms of actual value after excluding the insignificant terms was given below in equation. Final equation in terms of coded factors:

Biodiesel yield = $+3.97-0.083*A+0.068*B+0.060*C-0.024*D+0.031*A*B+0.051*B*C-0.21*A^2$

Where

A = Reaction temperature

B = Methanol to filter mud oil ratio

C = Catalyst to filter mud oil ratio

D = RPM

As shown the table 4.9 above, the model F-value of 52.89 implies the model is significant. Value of "Prob> F" less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AB, BC, A2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Temperature effects on biodiesel yield

The rate of reaction and bases catalyzed biodiesel production was highly influenced by temperature. Increasing temperature leads to decrease in the viscosity of biodiesel, increase in mass transfer between the solid catalyst and liquid reaction mixture, and increases rate of penetration of the filter mud oil into the pore of the catalyst. Thus, temperature was important parameter for higher reaction rate and higher yield biodiesel.

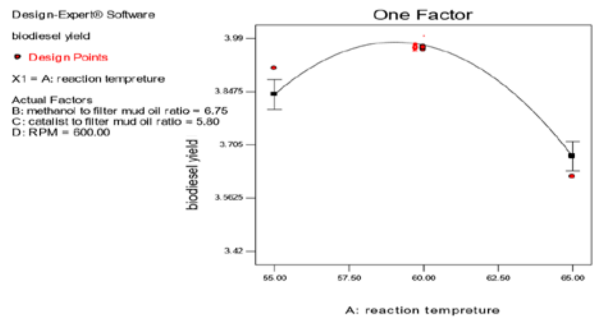

Figure 4.6 Effect of temperature on biodiesel yield at constant methanol to filter mud oil ratio and catalyst to filter mud oil ratio

The effect of temperature at constant methanol to filter mud oil ratio (6.75%) and the catalyst to filter mud oil weight ratio (5.8%) was studied. The figure shown above as the temperature increases from 55 to 60 oC, very small amount of increase in biodiesel yield seen. However, the yield has started to decline after 60oC. The reduction was significant after 60oC. The reason behind was as the temperature approaches the boiling point of methanol ,methanol start to appear in the vapor phase at the expense of available methanol for the reaction. As the temperature gets beyond the boiling point, significant methanol bubble formation was appeared, which inhibits the mass transfer process.

The effect of methanol to oil ratio on the biodiesel yield

The transesterification process consists of three consecutive reversible reactions. The triglyceride was successively transformed in to diglyceride, Monoglyceride and finally into fatty acid methyl ester (FAME) and glycerin. The ratio of methanol to filter mud oil ratio was one of the important factors that affect the conversion of triglyceride to biodiesel. Stoichiometrically, four mole of methanol are required for each mole of triglyceride, but in practice, a higher molar ratio was required in order to drive the reaction towards completion and produce more biodiesel as products. The results obtained in this study are in agreement with this, as shown the figure

(4.7) below, where at higher methanol to filter mud oil ratio, the biodiesel yield was increased. Higher ratio of methanol used could also minimize the contact of accesses triglyceride molecules on the catalyst active sites which could decrease the catalyst activity. Besides that, an increase in ratio of methanol could also lead to increase in the purity of the biodiesel layer which would also be responsible for the observed increase the in biodiesel yield.

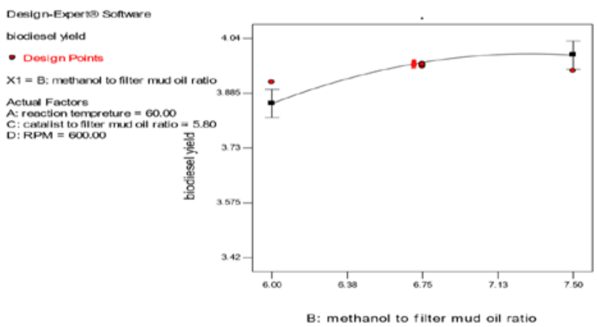

Figure 4.7 Effect of methanol to filter mud oil ratio on the biodiesel yield at constant temperature and catalyst to filter mud oil ratio

From the above figure, effects of methanol on the FAME yield was studied at constant temperature and catalyst to filter mud oil weight ratio 60oC and 5.8, respectively as shown the figure above. This was indicating that increasing of methanol to filter mud oil ratio from 6 to 7.5, increasing FAME yield from 3.36 to 3.97g. However, further increase doesn't have significant impact on the yield. Methanol to filter mud oil of 6.75 results 3.97g FAME yield. In addition, most probably at higher amount of methanol, the reverse reaction which was recombination of glycerol and Ester may had started to takes place, this in turn can leads to formation of di and Monoglyceride, which ends up with incomplete conversion.

Effects of catalyst to filter mud oil weight on the biodiesel yield

The amount of catalyst was important process variables for the production of biodiesel. In this experiment, catalyst to filter mud oil weight ratio was varied from 4 to 7.6 at constant temperature and methanol to filter mud oil ratio. The addition of more sodium hydroxide catalyst compensated for higher acidity, but the resulting soaps caused an increase in viscosity or formation of gels and interfered with separation of glycerol and contribute to emulsion formation during the water wash. When the active site of the solid catalyst was increased; accelerating transesterification reaction and increasing biodiesel yield. The Figure shows that increasing of the catalyst amount has significant positive effect on biodiesel yield. It was observed that when the catalyst does is low, incomplete transesterification was occurred. However, as the amount increases, more triglyceride was changed to ester. The reason behind was increasing of catalyst provides more active sites for the reaction of available triglyceride and methanol. In addition, the amount of available catalytic active surface was one important parameter to shift the reaction equilibrium forward in addition to the amount of methanol in the reaction mixture.

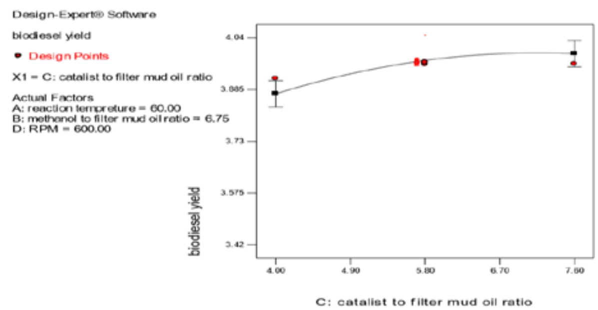

Figure 4.8 Biodiesel yield verses catalyst to filter mud oil weight

Characterization of biodiesel

The biodiesel obtained through transesterification process was characterized to know the fuel properties. The characterization of the biodiesel that produced from the filter mud oil were conducted from experimental analysis. The quality of biodiesel was very important for the performance and emission characteristics of a diesel engine. The characterization was performed for the five properties namely saponification value, acid value, flash point and kinematic viscosity. These properties were chosen for these main reasons. One because they are the defining properties of biodiesel and the other is because of the limitation of resource to perform other tests.

No	Properties	Unit	Value
1	Acid value	mgNaOH/g	0.0053
2	Saponification value	mgNaOH/g	97 – 124
3	Density	kg/m3	880.2
4	Dynamic viscosity	mpa	18.825
5	Kinematic viscosity	m2/s mm2/s	$2.1387x10^{-5}$ 21.38718
6	Higher heating	MJ/kg	44.182

	value		
7	Cetane number		81.15
8	Flash point	^{o}C	574.38
9			

From this result the density of the biodiesel was 880.2kg/m3, which are between the literature density (ASTM D6751 for biodiesel 870–890 kg/m3). Therefore the density of the biodiesel was acceptable.

4. CONCLUSIONS

1. Filter mud was shown to be a potential and promising feedstock for biodiesel production, in Ethiopia, based on its filter mud oil content and biodiesel yield.
2. Biodiesel was produced using methanol as a solvent and sodium hydroxide pellet as catalyst with a reaction time of 2, 4, 6, and 8 hours and the rate of mixing of 500, 600, and 700rpm at atmospheric pressure.
3.

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AUTHORS' SEMBLANCE

Pedro Dionisio Remedios- Castañeiras. Mechanical engineer, master in chemical engineering and doctor in technical sciences. Professor and collaborating researcher at the Center for the Study of Energy and Technological Processes at the University of Las Tunas, Cuba. Professor of unit operations of chemical processes. His lines of research are: fluid mechanics and pumping systems, viscous flows in the sugar industry, process energy efficiency and renewable energy. Current position as Full Professor, Thermodynamics, Renewable energy technology at Automotive Department of Federal Technical Vocational Education & Training Institute (TVETI), Addis Ababa, Ethiopia.

Abowerk Wagank: is an Ethiopian Engineer who joined Jimma University, Ethiopia, three year before. After completion of his bachelor degree in 2005 He enrolled for his master course in Faculty of Mechanical Engineering Jimma Institute of Technology. He is currently working as professor in Jimma University University.

Hailemariam Nigus: is an Ethiopian Engineer who joined Federal Technical Vocational Education and Training Institute (FTVET) Addis Ababa, Ethiopia, recently. He enrolled Adama University for his undergraduate study in 2003. After completion of his bachelor degree in 2008 he went to abroad for his MSc. Study to Tianjin University of Technology and Education, China and continued his study in mechanical design for three consecutive years. He completed his study with recognized achievement. He returns and works on the Institute as a lecturer. He is currently doing his PhD in mechanical design in Addis Ababa University.