A Comparative Study of Catalyst Effectiveness of Calcined Chicken Eggshells and Cowbone Ash in the Synthesis of Biodiesel from Chicken Fat and Pork Lard

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Abstract: This research was a comparative study of waste catalyst effectiveness in the synthesis of biodiesel of waste animal fats. The waste catalysts used were chicken eggshell and cow-bone ash, and the waste animal fats used were chicken fat and pork lard. Catalysts were prepared through calcinations at 900°C in a furnace to produce calcium oxide active sites. Eighteen transesterification experiments were carried out; nine experiments on chicken fat and nine experiments on pork fat. Reaction conditions for each experiment were 1:13 molar ratio of methanol, 5hr reaction time, 60-65°C reaction temperature. Chicken eggshell and cow-bone ash were varied at catalyst loading 5%wt, 7.5wt%, and 10wt% for both chicken fat and pork lard. Comparing starting materials, pork lard and chicken fat, data results showed that pork lard produced higher fatty acid methyl esters (FAME) yields on both chicken eggshell and cowbone ash catalysts; PLME yields were 81% and 82% compared to CFME yields of 74% and 78%. Pork lard proved to be best starting material to synthesize FAMEs using eggshell or cowbone catalysts. In economic terms for the industrial production of biodiesel from waste animal fats, catalysts are to be selected based on cost and availability. Eggshells produce better quality biodiesel over cow-bone ash based on results from viscosity, cloud point, and pour point analysis.

Keywords: biodiesel, chicken eggshell, cowbone, FAME, esterification, transesterification, GCMS

INTRODUCTION

Biodiesel is an alternative energy source that would combat the over-dependence on crude oil energy. With issues arising on the depletion of crude oil and its impacts on the environment, there is a need to develop alternative and sustainable means of energy. Biodiesel production would be a sustainable means of energy through the use of alternative low-cost raw materials that have less negative environmental impacts of global warming from crude oil. Biodiesel production has been hampered due to the high cost of production that leads to uncompetitive economic commodity and the food vs energy debate on biodiesel production negatively affecting the food industry. In light of these developments, there is research into using lowcost waste materials as raw materials in biodiesel production in order to increase its economic competitiveness, and to have a sustainable impact on the food industry through the recycling of waste materials. The main component of biodiesel is fatty-acid methyl esters (FAMEs). FAMEs can be produced from transesterification of lipids (fats and oils) in the presence of methanol and a catalyst. Waste animal fats such as chicken fat and pork lard provide the lowcost raw materials used in biodiesel production. Catalysts are also derived from waste materials such as eggshells and cow bone. These catalysts are calcined to produce calcium oxide which is the active catalyst in heterogeneous catalyzed transesterification. Comparative study of the waste catalysts and waste materials in maximum biodiesel yield production can be carried out to produce the most economically competitive biodiesel.

Experimental Procedures

MATERIALS AND METHODS

Waste Animal Fats

Waste animal fats (WAFs) contain mainly triacyglycerols used in the production of fatty acid alkyl esters. Vegetable oils are commonly used in biodiesel production because of their higher quality biodiesel yield and low pretreatment requirements; however, vegetable oils are economically uncompetitive due to high cost of production in comparison to low-cost waste animal fat feedstock.¹ This makes waste animal fats as most desirable feedstock in increasing the economic competitiveness of biodiesel. Furthermore, the undesirable nature of animal fat in human consumption makes waste animal fats suitable and sustainable feedstock in comparison to vegetable oils on the food vs energy debate. Waste animal fats are obtained from large meat processing facilities that would dispose of this unwanted animal parts; this provides the opportunity to tackle disposal problems as the waste animal fats are recycled into feedstock for biodiesel production, and the WAFs are very cheap raw materials. Most commonly used WAFs in biodiesel production are beef tallow from cattle, mutton tallow from sheep, and pork lard from rendered pork fat, chicken fat and animal grease. WAFs are characteristic for their high amount of saturated fatty acids (SFAs) which are a sum of the myristic, palmitic and stearic acid contents of the waste animal fat.^{1,2} Fuel properties of biodiesel are mostly determined by its fatty acid composition and the oil/fat quality. These fuel properties of biodiesel produced from WAFs are laid out in comparison to major parameters of petroleum-

based fuels. Provisional standards of biodiesel have been laid out to carry out comparative analysis of biodiesel from WAFs and petroleum-based fuels; the major parameters for comparison are density, kinematic viscosity, flash point, cetane number, and cold-filter plugging point.² Table 1.1 below shows the biodiesel standard adopted from the EN14214:

Property, Unit	EN14214 limits
	Min/max
Fame content %	96.5min
Density at 15°C, Kg/m ³	860/900
Viscosity at 40°C, mm ² /s	3.50/5.00
Flash point, °C	101 min
Acid Value mg KOH/g	6.0 min
Cetane number	51 min
Iodine value g I ₂ /100g	0.5
Cloud point, °C	5.0 max
Pour point, °C	4.0 max

Table 1: EN14214 Standard Biodiesel Properties

Pork Lard

Pork lard is obtained from pig slaughterhouses as rendered pork fat. Pork fat is a low-quality feedstock for biodiesel production due to its high free fatty acid (FFA) content. High free fatty acids and moisture (water) content could cause serious technical difficulties during biodiesel production leading to poor quality and impure biodiesel products that do not meet provisional standards.^{3,9} Below (Table 1.2) is the fatty acid profile of pork lard;

Fatty acids	Pork lard (wt %)	
Palmitoleic (C16:1)	2.2	
Palmitic (C16:0)	23.7	
Stearic (C18:0)	12.9	
Oleic (C18:1)	41.4	
Linoleic (C18:2)	15.0	
Linolenic (C18:3)	1.0	
Myristic (C14:0)	1.3	
Heptadecanoic (C17:1)	0.4	
Archidic (C20:0)	0.2	
Eicosenoic (C20:1)	0.9	
Eicosadienoic (C20:2)	0.7	
Eicosatrienoic (C20:3)	0.2	

Table 2: Fatty Acid Profile of Pork Lard

Chicken Fat

Chicken fat is obtained from poultry farm slaughterhouses. Chicken is the most consumed animal meat in the world. Due to the undesirable nature of chicken fat, there is great potential for chicken fat as feedstock material in biodiesel production. However, chicken fat usually requires pretreatment through esterification before transesterification in order to reduce the acid number below 1; a high acid value of more than 1 would result in the production of soap during the transesterification process.^{3,10} Below (Table 1.3) is the fatty acid profile of chicken fat.

Fatty acids	Chicken fat (wt %)	
Palmitoleic (C16:1)	7.7	
Palmitic (C16:0)	21.0	
Stearic (C18:0)	5.5	
Oleic (C18:1)	48.5	
Linoleic (18:2)	17.3	
Linolenic	0.0	
Unsaturated fatty acids	73.5	

Table 3: Fatty Acid Profile of Chicken Fat

Chicken Eggshell

The main composition of chicken eggshells is calcium carbonate (CaCO₃). Upon calcination at 900°C for 5hrs, the CaCO₃ decomposes to yield the active catalyst CaO, and carbon dioxide. The active CaO catalyst is stored in air-tight container to prevent deactivation. Chicken eggshells can be obtained from poultry farms. Chicken eggshells serve as low-cost and environmentally friendly catalysts in biodiesel production.^{1,5}

Cowbone

Cow bones are mainly composed of calcium carbonate CaCO₃ that are calcined at 900°C for 5hrs to yield CaO and CO₂. The active catalyst from calcination is CaO, a basic heterogeneous catalyst. The availability of cow-bone makes it a cheap and environmentally friendly material for catalyst in biodiesel production. ^{5,7}

Effect of Calcined Wastes

The optimal calcination temperature of animal bones was 900°C; at above this temperature, surface area, and thereby catalyst activity, was reduced. Calcination of animal bones at 900°C produced highly crystalline and active catalyst sites that yielded 96% FAME conversion.^{6,3} Catalyst loading on jatropha oil was varied from 2-6 wt %, and results showed that 6% produced maximum biodiesel yield of 96%. At above 6%, biodiesel yield dropped significantly.⁷ The transesterification of jatropha oil was carried for 3 hrs at 70°C using potassium hydroxide supported on calcined animal bones with varying amounts of methanol to oil ratio. Transesterification reactions require excess methanol to drive reaction in forward direction because transesterification reactions were reversible reactions. The optimal conditions for jatropha oil conversion to 96% were 6 wt% catalyst loading, 70°C reaction

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temperature, and 9:1 methanol to oil ratio.⁷ Calcined eggshells were used in transesterification of palm oil into biodiesel of 97-98% FAME yield using optimal parameters of 10 wt% catalyst loading, 65°C reaction temperature, 9:1 methanol to oil ratio. The high biodiesel yield showed the high effectiveness of calcined waste eggshells in converting palm oil to methyl esters.^{7,2} Chicken eggshells contain 85-95% calcium carbonate which makes them good sources for calcium oxide. In laboratory scale experiments comparable to CaO synthesized from other sources, transesterification reactions of 3hr reaction time produced 95% FAME yield using calcined chicken eggshells. The following results were also generated using calcined chicken eggshells; chicken eggshell calcined at 800^oC for 2-4 hrs used in a transesterification reaction with parameters of 60^oC reaction temperature, 12:1 methanol to oil ratio (molar ratio), 10% wt (weight/weight) of catalyst, and 2 hr reaction time yielded 95% FAMEs; chicken eggshell calcined at 1000°C for 2 hrs used in transesterification reaction with parameters of 65°C reaction temperature, 9:1 methanol to oil ratio, 3% wt of catalyst, and a 3 hrs reaction time yielded 95% FAMEs; Chicken eggshell calcined at 900^oC for 2 hrs used in a transesterification reaction with parameters of 60C reaction temperature, 9:1 methanol to oil ratio, 3% wt of catalyst, and 3hr reaction time vielded 96% FAMEs.^{8,6} These reactions were conducted at varying reaction parameters and the results showed high yields or conversion into FAMEs. However, the transesterification reactions were conducted on various feedstock materials; this research focuses on combating the economic non-competitiveness of biodiesel by specifically focusing on catalyst activity on cheap sources such as chicken fat and pork lard. Kirubakaran et al noted that methanol to oil ratio was the most significant factor in biodiesel yield with optimum conditions for transesterification as follows; 8.5wt% catalyst loading, 57.5°C reaction temperature, 1:13 methanol to oil ratio, 5 hrs reaction time. This produced a biodiesel yield of 90.41%.^{9,5} Dias et al noted that calcium oxide as the most promising catalyst with favorable reaction conditions, lower solubility, highly active basic sites, and cheap cost.^{10,3} The above literature review showed the highly effective calcium oxide catalyst from calcined wastes. Transesterification reactions were carried out on various feedstock materials to produce high yields of above 90% FAME conversion. This research focuses on increasing the economic competitiveness of biodiesel by using cheap raw material sources such as chicken fat, pork lard, and eggshell and cowbone for catalysis. A comparative study on catalyst effectiveness of catalyst loading in maximum biodiesel yield from chicken fat and pork lard is the focus of this research.

Experimental Procedure.

Oil Extraction

Chicken fat and Pork lard were placed in a microwave to extract oil from the fat. The oil was centrifuged at 6000rpm for 10 minutes to remove particulate matter and impurities. The extracted oil was dried in an oven at 100°C for 10 minutes.

Acid Value Test

Acid value test was carried out to determine the free fatty acid content or acid value of the extracted oil; an acid value above 1 would result in the production of soap during the

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transesterification reaction. From literature review, it was seen that acid value of pork lard and chicken fat could be above or below 1 depending on the fat obtained. Acid value tests from literature review indicated that sample sizes are an important factor in accurately determining the acid value. If the expected acid value was expected to be below 1, 20 grams of oil was used. If the expected acid value was expected to be in the range of 1-4, a sample test size of 5-10 grams was used; 10 grams was used in this experiment. The solvent mixture was prepared by mixing equal portions of isopropyl alcohol and toluene to make 50ml of solvent mixture. The oil was mixed with the solvent and drops of phenolphthalein were added. The solution was titrated with a potassium hydroxide in ethanol solution until a pink color change was observed. The formula $\frac{56.1 X (A-B)X 0.1}{Mass of Oil}$ was used to calculate the acid value of the oil where A is the volume of KOH used and B is correction for blank.

Esterification

Chicken fat produced an acid value above 1 for each sample test size used. This meant that chicken fat had to undergo esterification before transesterification in order to reduce the acid value, and hence, reduce soap formation during transesterification. Literature review showed that in earlier research works sulfuric acid was used as catalyst in esterification of chicken fat, however, a recently published work used hydrochloric acid as catalyst. In order to determine which catalyst was most suitable with respect to acid value, a preliminary test was carried out using both catalysts. Sulfuric acid proved to be the more efficient catalyst producing a lower acid value on chicken fat than hydrochloric acid. The esterification reaction conditions were set as follows; 6:1 methanol to oil ratio, 3 wt% catalyst loading, 60-65°C reaction temperature, 1 hr reaction time (this reaction conditions were used in the preliminary tests using sulfuric acid and hydrochloric acid catalysts). The esterification products poured into a separating funnel and allowed to separate overnight. The oil was decanted and stored for transesterification.

Transesterification

A total of 18 experiments were carried out, 9 experiments on chicken fat and 9 experiments on pork lard. Reaction Conditions for each experiment were as follows; 13:1 methanol to oil ratio (molar ratio), 60-65°C reaction temperature, 5 hrs reaction time. Chicken eggshell and cowbone ash were varied at catalyst loading 5% wt, 7.5wt%, and 10wt% for both chicken fat and pork lard. Each catalyst loading was repeated 3 times to obtain consistent results. After transesterification for 5hrs, the mixture was allowed to cool and centrifuged at 6000rpm for 10 minutes. The centrifuged mixture was poured into a separating funnel to separate the methanol from biodiesel. Methanol was above while the biodiesel was below; this was verified through GCMS analysis. The separation was allowed for at least 15hrs, and the biodiesel was collected. The biodiesel was heated above 70°C to remove excess methanol. The percentage yield of biodiesel was calculated from the mass of oil used in the reaction (indicated in the color red and % in the results flowchart). The biodiesel was stored for further analysis. The equation for the transesterification reaction is shown in Figure 1 below:

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Figure 1: Transesterification reaction

Gas Chromatography and Mass Spectrometry

GCMS analysis was carried out on each catalyst to waste animal fat combination to determine if the product was a fatty-acid methyl ester (FAME). Samples were put into the GCMS machine and the parameters for analysis were set. The gas chromatography separates the sample into individual components, and the mass spectroscopy characterizes the individual components. The chromatogram of the sample was produced on the results screen and the most intense (most abundant) peak was selected to produce the mass spec of the peak. The mass spectroscopy of the peak was run through a library of mass spectra of various compounds in order to get the desired match of fatty-acid methyl esters.

Viscosity Analysis

Viscosity is a measure of the resistance of a fluid to flow. The EN14214 has standards for biodiesel products that can be practically applied into diesel engines, and these standards must be met in order to ensure the use of the biodiesel to run these diesel engines. Viscosity analysis was carried out using a viscometer placed in a water bath of 40°C, and a stopwatch to measure time. The experiment was carried out twice on each catalyst loading result to determine the viscosity of the biodiesel product.

Cloud and Pour Point Analysis

Cloud and pour point analysis is carried out to determine the behavior of the biodiesel in colder regions of the earth; it is done to know if the biodiesel would still be usable in diesel engines when the temperature is relatively low and nearing sub-zero levels. The EN14214 has standards for the cloud and pour point of a biodiesel product that must be met in order to ensure efficient operation of the diesel engine in colder regions or temperatures. The samples were analyzed in a cloud and pour point machine and the results were collected.

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RESULTS

Acid Value Test

Table 4: Pork Lard Acid Value Test

Trial	Mass of Oil (g)	Volume of KOH	Blank Correction	Acid Value
		(ml)		
1	20	3.42	0.4	0.84711
2	20	3.5	0.4	0.86955
3	20	3.47	0.4	0.861135
4	10	1.7	0.4	0.7293
5	10	1.8	0.4	0.7854
6	10	1.76	0.4	0.76296

Table 5: Chicken Fat Acid Value Test

Trial	Mass of Oil (g)	Volume of KOH	Blank	Acid Value
	_	(ml)	Correction	
1	20	5.5	1	1.26225
2	20	5.8	1	1.3464
3	20	5.76	1	1.33518
4	10	3.65	1	1.48655
5	10	3.6	1	1.4586
6	10	3.55	1	1.43055



Figure 2: Acid Value Chart of Pork Lard and Chicken Fat

The bar chart above for the acid value showed that chicken fat had an acid value above 1 and therefore had to undergo esterification before transesterification. Although some reports from literature review showed that chicken fat had an acid value of 1, the FFA content of chicken fat is entirely dependent on the fat obtained. Acid value tests have to be carried out to accurately determine the acid value of chicken fat as FFA content may vary from one chicken fat to the other. Pork lard FFA content on all literature review reports showed that the FFA content was always less than 1, and always less than that of chicken fat. Transesterification

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Scheme 1: Flowchart of Chicken Eggshell/Pork Lard Transesterification



Scheme 2: Flowchart of Chicken Eggshell/ Chicken Fat Transesterificatio

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Scheme 4: Flowchart of Cow-bone ash/Chicken fat Transesterification

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Figure 3: Percentage Yield of Pork Lard Methyl Esters with Egg-shell Catalyst

The chart above shows the percentage yield of pork lard methyl esters from chicken eggshell catalysts. The best catalyst loading for pork lard and chicken eggshell catalyst is 5%.



Figure 4: Percentage Yield of Pork Lard Methyl Esters with Cow-bone Ash Catalyst

The chart above shows the percentage yield of pork lard methyl esters (PLME) using cow-bone ash as catalyst. The best catalyst loading for cow-bone ash and pork lard is 10% Comparison

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of cow-bone ash catalyst to eggshell catalyst (Table 6) based on percentage yield of pork lard methyl esters (PLME), it showed that both catalysts can be used.

Table 6: Comparison of Catalyst PLME Yield

Catalyst	Loading	PLME Yield
Eggshell	5%	81%
Cowbone Ash	10%	82%

Both catalysts produce very similar percentage yield of PLME. However, eggshells are more efficient catalysts due to smaller loading percentage produces high PLME yield when compared to cow-bone ash. Economic implications of cost and availability of both catalysts have to be considered when choosing catalyst for PLME production. Chicken eggshell is the best option because it is easily obtained and much easier to handle. Also, eggshells are cheaper than cow-bones, and more readily available.



Figure 5: Percentage Yield of Chicken Fat Methyl Esters with Eggshell Catalyst

The chart above shows the percentage yield of chicken fat methyl ester (CFME) using chicken eggshell catalysts. 5% and 10% catalyst loading show very similar percentage yield but, 5% is the best due to having less standard deviation as reflected in the error bars.

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Figure 6: Percentage Yield of Chicken Fat Methyl Esters with Bone-Ash Catalysts

This chart shows percentage yield of chicken fat methyl esters (CFME) using cow-bone ash catalysts. 5% is the best catalyst loading for CFME yield using chicken eggshell. Comparison of chicken eggshell and cow-bone ash catalysts in CFME yield (Table 6) showed that cow-bone ash was the best catalyst for CFME yield.

Table 6:	Comparison	of Catalyst	CFME	Yield
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Catalyst	Loading	Yield
Cowbone Ash	5%	78%
Cowbone Ash	7.5%	74%

Comparing starting materials, pork lard and chicken fat, data results showed that pork lard produced higher fatty acid methyl esters (FAME) yields on both chicken eggshell and cowbone ash catalysts; PLME yields were 81% and 82% compared to CFME yields of 74% and 78%. Also, the economic implications of esterification before transesterification must be considered. Chicken fat most always has an acid value of above 1 in comparison to pork lard, and therefore must undergo esterification before transesterification. Given this, pork lard should be the preferred waste animal fat for biodiesel production. However, availability can become an issue. It can be argued that chicken fat is readily more available than pork lard. If the availability of chicken fat and its esterification costs outweigh the availability of pork lard in economic terms, then chicken fat would be a better starting material.

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Gas Chromatography and Mass Spectrometry (GCMS)

GCMS analysis was carried out on each biodiesel product to verify that the product was indeed a fatty acid methyl-ester (FAME). The results of GCMS analysis are below:



Figure 7: Chromatogram of Chicken Fat and Cow-bone Ash



This is unknown spectrum of the most abundant peak at 9.937 minutes of chicken fat and cowbone ash biodiesel analysis.



Figure 9: Mass Spectrum of 10-Octadecanoic acid Methyl Ester



Figure 10: Chromatogram of Chicken Fat and Chicken Eggshell In comparison to the unknown spectrum above, it shows that the spectrum is a 10-octadeneoic acid methyl ester, a fatty acid methyl ester.



This is the unknown spectrum at the most abundant peak at 9.856 mins of chicken fat and chicken eggshell biodiesel.

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Figure 12: Mass Spectrum of 10-Octadecanoic acid Methyl Ester

The peak spectrum matches a fatty acid methyl ester.



Figure 13: Chromatogram of Pork Lard and Cow-bone Ash



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This is unknown spectrum for the most abundant peak at 9.966 mins of pork lard and cowbone ash biodiesel.



Figure 15: Mass Spectrum of 10-Octadecanoic acid Methyl Ester The peak matches a fatty acid methyl ester.









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This is the spectrum of the most abundant peak at 9.963 min of the pork lard and chicken eggshell biodiesel.



The peak matches a fatty acid methyl ester.

Viscosity Analysis Results Table 7: Viscosity Tests

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ID	Trial 1 (s)	Trial 2 (s)	Mean Value	Viscosity (cSt)
5%CF	6.49	6.47	6.48	18.26064
5%PF	5.54	5.66	5.6	15.7808
7.5%CF	1.94	1.81	1.875	5.28375
7.5%PF	1.66	1.66	1.66	4.67788
10%CF	2.47	2.47	2.47	6.96046
10%PF	1.78	1.81	1.795	5.05831
5%AF	7.94	8.1	8.02	22.60036
5%BF	11.62	11.53	11.575	32.61835
7.5%AF	10.25	10.34	10.295	29.01131
7.5%BF	10.56	10.66	10.61	29.89898
10%AF	8.72	8.65	8.685	24.47433
10%BF	8.82	8.82	8.82	24.85476

(CF= Chicken fat + Chicken eggshell, PF= Pork lard + Chicken eggshell) (AF= Chicken Fat + Cow-bone ash, BF= Pork lard + Cow-bone ash)





The viscosity bar chart (Figure 19) shows that chicken eggshells catalysts on both pork lard and chicken fat produce better quality biodiesel with regards to the FAME content. In comparison to the EN14214 standard biodiesel properties, viscosity measured at 40°C should have a minimum value of 3.50 and a maximum value of 5.00. Results of chicken eggshell and pork lard have a 4.68 value which meets the required EN14214 standard. Other values close to the required standard show that chicken eggshell catalyst produce better quality biodiesel when compared to cow-bone ash.

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Cloud and Pour Point Analysis Results Table 8: Cloud and Pour Point Tests

CLOUD POINT		POUR POINT	
ID	CP (oC)	1D	PP (oC)
5%CF	18	5%CF	15
7.5%CF	9	7.5%CF	0
10%CF	4	10%CF	3
5%PF	8	5%PF	-3
7.5%PF	4	7.5%PF	3
10%PF	3	10%PF	3
5%AF	13	5%AF	0
7.5%AF	9	7.5%AF	-3
10%AF	14	10%AF	6
5%BF	20	5%BF	15
7.5%BF	25	7.5%BF	24
10%BF	28	10%BF	24



Figure 20: Cloud and Pour Point Chart

A similar trend is observed in the cloud and pour point chart. EN14214 standard biodiesel properties require cloud point of 5.00 maximum value and pour point of 4.00 maximum value. Chicken eggshells produce the better-quality biodiesel on both pork lard and chicken fat that meet the EN14214 standards. It is also observed that at 7.5% and 10% catalyst loading of chicken eggshell produce better quality biodiesel based on viscosity analysis, and cloud and pour point analysis.

CONCLUSION

On comparative analysis, pork lard is the best starting material for synthesis of fatty-acid methyl esters (FAMEs) using both eggshell and cowbone ash catalysts. This is because of the tendency for chicken fat to have a higher FFA, and would incur economic costs for esterification to drop the acid value. The most suitable catalyst for transesterification is selected based on cost and availability; eggshell is most suitable due to cheaper source, availability and easy handling. Furthermore, eggshell catalysts produce higher quality biodiesels which is seen in the viscosity, cloud and pour point analyses. FAME content and GCMS quantification to accurately estimate the biodiesel quality were not covered in the scope of this research.

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