



Presentation on

Preparation and characterization of biodiesel from Karanja oil by using silica gel reactor

Presented by

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Overview

- Purpose
- Introduction
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Purpose of this work

“To study the effect of silica gel as a desiccant in the conversion reaction for biodiesel preparation from raw oil and the optimum dosing of silica gel.”



Introduction

- ❖ The world energy demand is increasing gradually which also leads to increase environmental pollution. Due to these reasons energy engineers are searching new, noble, and renewable energy resources to meet the increasing energy demand.
- ❖ Biodiesel from vegetable oil is one of the most efficient forms of renewable energy which can be alternative to the fossil fuel.



Introduction (*continued*)

- ❖ Biodiesels are renewable, biodegradable and has lower emissions compared to petroleum diesel.
- ❖ Methyl ester of karanja oil has been investigated as an alternative renewable fuel.
- ❖ Esterification of carboxylic acids, especially direct esterification between carboxylic acids and alcohols has wide academic as well as industrial applications .



Introduction (*continued*)

- ❖ In esterification reaction due to methanolysis little amount of water formed and another little amount of water may remain with the converted free fatty acid (FFA).
- ❖ De-touching the unwanted water or moisture by silica gel from the conversion better biodiesel can be prepared.
- ❖ The most economical way for biodiesel conversion by using silica gel has been investigated in this work.



Materials and Methods

Chemicals:

- silica gel 60 (0.040-0.063 mm)
- sodium hydroxide (97%)
- hydrochloric acid (37%)
- sulfuric acid (98%)
- phenolphthalein (reagent grade *pH* 8.2-9.8)
- diethyl ether (96-98%)
- methanol (99.8%)
- ethanol (99%)
- glacial acetic acid
- Chloroform
- potassium iodide
- Iodine and
- karanja oil.



Materials and Methods (*continued*)

Three step method:

- ❖ saponification, acidification and esterification were carried out .
- ❖ The calculated amounts of sodium hydroxide for saponification, hydrochloric acid for acidification, sulfuric acid and methanol for esterification were added.
- ❖ *In saponification*, 40 ml karanja oil was mixed with required aqueous sodium hydroxide solution in a 500 ml three necked flask. Then the reaction was carried out at 100°C under reflux with vigorous stirring (550 rpm) for two hours.



Materials and Methods (*continued*)

- ❖ Then the reaction is stopped and the saponification product was cooled to 60°C.
- ❖ *In acidification*, required amount of hydrochloric acid was added slowly to the soap solution and the reaction is done at 60°C with stirring until the soap is dissolved fully.
- ❖ Then it was transferred to the separatory funnel and given hot water wash to remove mineral acid. FFA (upper layer) was separated and dried by vacuum distillation.



Materials and Methods (continued)

- ❖ Finally it's FFA content was measured by titrametric method.
- ❖ *In esterification*, the above product is kept in a 250 ml three necked flask and metho-sulfonic acid solution was added to that. Metho-sulfonic acid solution is a mixture of required amount of methanol and sulfuric acid.
- ❖ The reaction runs with different temperatures with 550 rpm stirring under reflux condensation for various reaction times. In esterification reaction silica gel was used to adsorb water produced in esterification reaction, hence increase the reaction rate.



Fig. 1 Experimental setup for the biodiesel production using three-step acid catalyzed method .



Materials and Methods (*continued*)

- ❖ The molar ratio of FFA to methanol, catalyst concentration and reaction temperature were optimized.
- ❖ Then the product is cooled to room temperature, transferred to separatory funnel, the biodiesel product was washed by hot deionized water several times until the washing became clear and the upper layer (biodiesel) is separated after half hour.
- ❖ The product was filtered and then distilled under vacuum to remove moisture at 100°C and then biodiesel properties are measured.
- ❖ Using the optimum conditions one biodiesel sample was prepared and properties were measured.



Materials and Methods (continued)



(a)



(b)



(c)



(d)

Figure 2: (a) Karanja seed kernel (b) Silica gel (c) Experimental setup for the biodiesel production using three-step catalyzed method using silica gel and (d) Produced biodiesel.



Materials and Methods (*continued*)

Three step method with silica gel

- ❖ It's almost similar with the three step method described before.
- ❖ Here just at the starting of the esterification 5 gm dried silica gel (silica gel: oil=1:5) was added to the esterification mixture.
- ❖ properties of this bio-diesel were measured by maintaining different ASTM standards.

Materials and Methods (*continued*)



Effect of silica gel in esterification reaction

Table 1. Preparation of biodiesel with different dosing of silica gel in esterification.

Observation	First Step	Second Step	Third Step	Fourth Step
Dose 1	Initially (0 g SG)	After 20 min (3g SG)	After 20 min (2g SG) and continued to 140 min.	
Dose 2	After 20 min (5g SG) and continued to 160 min.			
Dose 3	After 10 min (2g SG)	Then after 10 min (2g SG)	Then after 10 min (1g SG) and continued to 150 min.	
Dose 4	After 5 min (2g SG)	Then after 5 min (2g SG)	Then after 5 min (1g SG) and continued to 165 min.	
Dose 5	Initially (1g SG)	Then after 5 min (2g SG)	After 5 min (2g SG) and continued to 170 min.	
Dose 6	Without silica gel the reaction continued to 180 min.			
Dose 7	Initially (5g SG) and continued to 180 min.			
Dose 8	Initially (5g SG)	After 20 min (2g SG)	Then after 20 min (2g SG)	Then after 20 min (1g SG) and continued to 120 min.
Dose 9	Initially (10g SG) and continued to 180 min.			
Dose 10	Initially (5g SG)	After 15 min (5g SG) and continued to 165 min.		



Result and Discussion

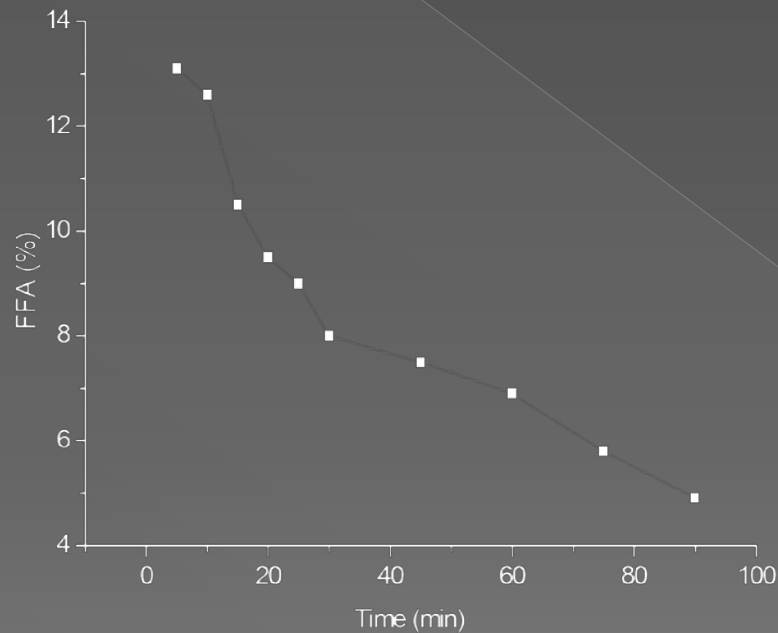
Three step method with and without silica gel

Table 2. Properties of biodiesel produced by three step method
(a) without silica gel and (b) with silica gel.

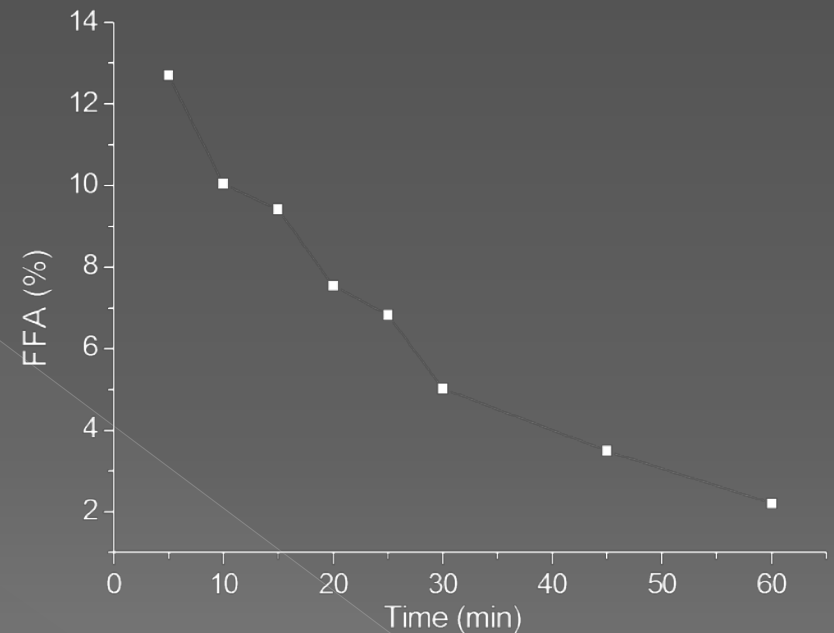
Property	(a) Measured value	(b) Measured value
Color	Radish-black	Radish-black
Viscosity (mm ² /s)	4.57	3.75
Saponification value (mg KOH/gm oil)	138	140
FFA (wt %)	4.91	2.20
Specific gravity	0.89	0.87



Result and Discussion (continued)



(a)



(b)

Figure 3. FFA reduction in Esterification reaction (a) without silica gel and (b) with silica gel [catalyst (H_2SO_4) concentration 5 wt% of FFA, Methanol/FFA= 9:1 molar ratio, vigorous stirring and reaction time 90 min at 60°C .]



Result and Discussion (continued)

Effect of amount of silica gel in esterification

Table 3: Properties of biodiesel produced after one hour esterification reaction with and without silica gel.

Amount of silica gel initially fed in esterification	FFA (%) of Bio-diesel	Viscosity (mm ² /s) of Bio-diesel
No silica gel	4.91	4.53
5 gm	3.32	4.01
10 gm	3.22	4.01



Result and Discussion (continued)

Table 4. Properties of biodiesel produced after one hour esterification reaction with and without silica gel at different time interval

Silica gel fed with the variation of time interval	FFA (%) Of Bio-diesel	Viscosity (mm ² /s) Of Bio-diesel
Dose 1	5.82	4.80
Dose 2	7.52	4.91
Dose 3	7.41	5.13
Dose 4	7.59	4.77
Dose 5	5.47	4.41
Dose 6	6.73	4.42
Dose 7	3.28	4.82
Dose 8	3.99	4.11
Dose 9	2.63	4.02
Dose 10	3.36	4.16



Summery

- ❖ Biodiesel prepared by three-step method (without silica gel) has 4.91% FFA content, 4.57 mm²/s viscosity.
- ❖ Biodiesel prepared by three-step (with silica gel) has 2.16% FFA, 3.75 mm²/s viscosity. Optimum silica gel, oil ratio is 1: 5 (wt/vol).
- ❖ In esterification reaction using silica gel initially is the best.
- ❖ The biodiesel preparation using silica gel results better properties within shorter time.



Summery (*continued*)

- ❖ Finally it can be concluded that the karanja biodiesel prepared by the three-step method with silica gel is better and suitable to use.
- ❖ Further study is needed in these fields for engine performance and emission study by using this biodiesel and their blends.



Comments?
Questions?
Observations?



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THANK YOU ALL