

Potential Heterogenous Catalyst from Pineapple Waste for Biodiesel Production

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ABSTRACT

Biodiesel is a viable alternative to petroleum-based fuels due to its potential to lower greenhouse gas emissions. Recently, the utilization of waste biomass materials for the production of fuels and other energy products has been explored. In this current study, a potential heterogeneous solid catalyst was obtained from pineapple waste peels and graphite oxide (GO). Its potential for transesterification in biodiesel production was investigated using the impregnation mass ratio of 1 GO: 3 pineapple waste. The methanol to coconut oil molar ratio of 18:1, impregnation temperature of 80 °C and H₃PO₄ activating agent were found to yield the highest fatty acid methyl ester (FAME) yield of 53.71%.

Keywords: Biodiesel, Transesterification, Heterogeneous Catalyst, Pineapple waste peels, optimization, Graphite Oxide.

INTRODUCTION

As countries develop and living standards improve, the world consumption and demand for energy will also inevitably and rapidly increase. The largest source of world energy consumption remains to be petroleum. In January 2017, petroleum consumption in the Philippines was reported by the Department of Energy to be 391.85 thousand barrels per day [1]. This continuous increase in consumption brought out environmental concerns as petroleum-based diesel fuel is considered as one of the prime contributors in the global warming. Increase in the emission of greenhouse gases into the atmosphere was due to the combustion of fossil fuel. It has been shown that 98% of carbon emissions are from fossil fuel combustion. In the Philippines, CO₂ emission was at level of 55 million metric tons in 2015, a change of 14.58 % from 48 million metric tons the previous year as shown in the Climate Transparency Report [2, 3].

Among the most promising fossil fuel alternatives, biodiesel is found to be effective because of its potential to reduce green house gas emissions. Biodiesel is proven to reduce overall CO₂emissions by 78% as opposed to petroleum-based diesel fuel [4]. Biodiesel represents a renewable and eco-friendly biofuel suitable for use in diesel engines either in its pure form or when blended with traditional diesel. It is derived from oil conversion through methods like pyrolysis, micro-emulsification, and most notably, transesterification. Transesterification is the leading process for producing biodiesel that is both cleaner and more environmentally friendly. Additionally, it is known for delivering the highest bioenergy output in a shorter timeframe compared to other conversion processes [5].

Catalysts synthesized from biomass are under intensive investigation as possible alternatives to traditional chemical catalysts for the sustainable production of biodiesel. This interest is due to their excellent characteristics: high catalytic activity, low cost, abundant availability, and eco-friendly and efficient manufacturing processes. [6]. Heterogeneous catalysts for biodiesel production from agricultural wastes have been developed in recent years because they are cost-effective, chemically safe and easily accessible [7]. Reported studies on the use of fruit peel wastes to derive heterogeneous catalyst with excellent catalytic activities include banana [8-9], tamarind [10], passion fruit [11,12], avocado [13], citrus [14], etc.

The purpose of this study is to prepare a heterogenous catalyst from the fruit peel of *Ananas comosus* commonly known as pineapple which is commercially cultured in the Philippines and other tropical countries. Previous studies indicated that *A. comosus* contains high carbon content which means that it is a good source of activated carbon [15]. Another study done on *A. comosus* was the utilization of its leaves ash as a catalyst for biodiesel production [16]. However, little research has previously been conducted on the utilization of a composite material (fruit peel activated carbon in combination with graphite oxide) as catalyst in biodiesel production. The structure and properties of graphite oxide vary based on the synthesis method and the extent of oxidation. While it generally retains the layered structure of the original graphite, its layers become buckled, and the interlayer spacing increases to roughly twice that of graphite, approximately 0.7 nm. [17]. However, the graphite oxide must be further modified through impregnation to increase its functionalization. The impregnation with graphite oxide is said to enhance the durability of the produced complex catalyst. Impregnation is a process to fill the pore of a carrier with an active phase. It is a commonly employed process for supported catalysts in which it includes filling the pores of the support with the catalyst precursor solution. This is to achieve the desired loading and to enhance the efficiency of a catalyst. According to Julkapli *et al.*, graphene tends to stack together via pi-to-pi interactions and enfoldment of graphene sheets and its small interlamellar spacing resulting to limiting its utilization. Thus, structural changes brought by impregnation with another chemical compound can be useful for extending the functionalization of graphene [18]. Hence this study was conducted to investigate its efficacy as well as to determine the optimized condition of certain specific reaction parameters.

EXPERIMENTAL

Materials

Pineapple peels were purchased outside the Bugo Cannery of Del Monte Philippines, Inc. Coconut oil was purchased from the local supermarket. HPLC-grade methanol (>99% purity, Scharlau) was procured from Merteflor. Graphite oxide was provided by Dr. Juliet Q Dalagan and Ms. Romelisa A Ibale. Other chemicals such as phosphoric acid and potassium hydroxide were supplied by the laboratory of Xavier University-Chemistry Department.

Approximately ten (10) grams of pineapple peel were immersed in 40 wt% KOH followed by heating in an oven for one hour until temperature was 80 °C. The same procedure was done using 20 wt% H₃PO₄ as the activating agent. The dried pineapple peel was heated for 4 hrs in a furnace until the temperature of 400 °C and 800°C for KOH and H₃PO₄ was achieved, respectively. The activated carbon was washed, dried 24 hours at 100 °C and stored in a desiccator. Impregnation method was done to support pineapple peel activated carbon (PPAC) with graphite oxide in a mass ratio of 1:3 by combining 1.5 mg of GO to 4.5 mg PPAC in 3 mL of water. Stirring for 4 hours at room temperature, at 50°C and at 80°C was employed followed by drying in an oven for 12 h at 100 °C.

The catalyst produced was characterized using FTIR (Perkin Elmer FT-IR 100 Spectrometer) from the scanning range of 4000–400 cm⁻¹ and Scanning Electron Microscopy (JEOL JSM 5310 SEM). Using

transesterification reaction, coconut oil was converted to FAME with varying methanol to oil molar ratio of 6:1, 12:1 and 18:1 and fixed catalyst loading of 1.5%, reaction temperature of 60°C and reaction time of 90 min. Vacuum filtration was used to isolate fatty acid methyl ester (FAME) followed by centrifugation. FAME composition was analyzed using Gas Chromatography with Flame Ionization Detection (HP-6890) with ultra-alloy as column and helium as the carrier gas.

RESULTS AND DISCUSSION

Carbonization and Chemical Activation

To produce activated carbon from pineapple peel wastes, phosphoric acid (H_3PO_4) and potassium hydroxide (KOH) were used as activating agents. The preparation included carbonization and activation using muffle furnace at 800°C for H_3PO_4 -treated raw pineapple peels and 400°C for potassium hydroxide. Chemical treatment caused changes in color from yellowish green to brown/black and changes in texture from stiff to tender. PPAC by phosphoric acid (H_3PO_4) have relatively higher yield ($27.76\% \pm 2.50$) than KOH-activated PPAC ($8.02\% \pm 2.34$). In the work of Basta et al., lower carbon yield using KOH could be attributed to the breakdown of biomass into volatile matters [19].

The surface morphology of the raw pineapple peel and the prepared activated carbon were examined using SEM. The SEM images in **Figure 1** showed a significant difference in the surface morphology of the raw sample without activation and the samples after activation with KOH and H_3PO_4 . Non-porous and rugged surface was observed for the raw pineapple peels (**Figure 1A**). In contrast, uneven cavities and fine open pores were seen on the surface of the AC obtained after the chemical treatment (**Figures 1B and 1C**). The formation of these cavities could be due to the release of volatiles from the raw-PP during carbonization [20].

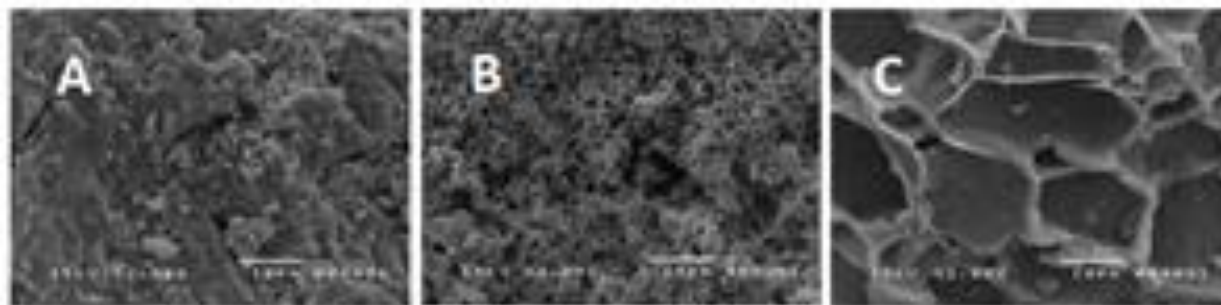


Fig. 1. SEM images of raw-PP (a), PPAC-KOH $T_{400^\circ C}$ (b) and PPAC- H_3PO_4 $T_{800^\circ C}$ (c) in 2,000x magnification

As for the SEM image of PPAC- H_3PO_4 $T_{800^\circ C}$ in **Figure 1C**, surface morphology was irregular like that of PPAC-KOH $T_{400^\circ C}$ but a clearer surface was observed. Ilomuanya *et al.* pointed out that phosphoric acid may be the reason for the unclogging of the micropores and mesopores by preventing the formation of tar [21]. The liberation of tar makes the surface more defined and finer. The porous structure illustrated in the SEM supports the possibility of AC as a catalyst modifier in which the pores could act as sites for adsorption.

Surface Functional Group Analysis

An overlay of the FTIR spectra is shown in **Figure 2** which verified the presence of the functional groups in the raw PP and prepared AC. In raw PP, the presence of $-OH$ stretching vibration at $3300-3400\text{ cm}^{-1}$ are indicators of the presence of alcohol, phenol, and carboxylic acid. The weak band at $2960-3000\text{ cm}^{-1}$ represents a C-H stretching-vibration while the weaker bands at $1260-1601\text{ cm}^{-1}$ can be attributed to C=C

aromatic ring stretching vibration. The strong peak at 1031 cm^{-1} can be due to C-O. A very low signal for C=O was observed at 1746 cm^{-1} which probably means an inadequacy in the number of esters, ketones, and aliphatic acids in the raw pineapple peels. The spectra for PPAC-KOH_{T400°C} and PPAC-H₃PO₄ T800°C showed same characteristic peaks. The prepared activated carbons demonstrate very weak absorbance for C-H stretching at 2993 cm^{-1} and 3000 cm^{-1} . The reduced intensity of bands between $1260\text{--}1601\text{ cm}^{-1}$ may indicate the bond cleavage of C=C stretch and the progressive elimination of hydrogen functional groups. PPAC-KOH_{T400°C} and PPAC-H₃PO₄ T800°C have lower intensity of C-O compared to raw-PP and both have no signal for C=O. The addition of activators at high temperature may have caused the degradation of organic material that weakened the surface structure of activated carbon and disappearance of some of the surface functional groups. Peaks slightly shifted towards higher wavenumber which can be linked to physical changes in the molecule. In FTIR spectra, shifting of peaks towards lower or higher wavenumber is related to mass of the molecule, with shifts to higher wavenumber corresponding to reduction in mass and vice versa. The changes may be due to internal or external effects of chemical activation on the molecule.

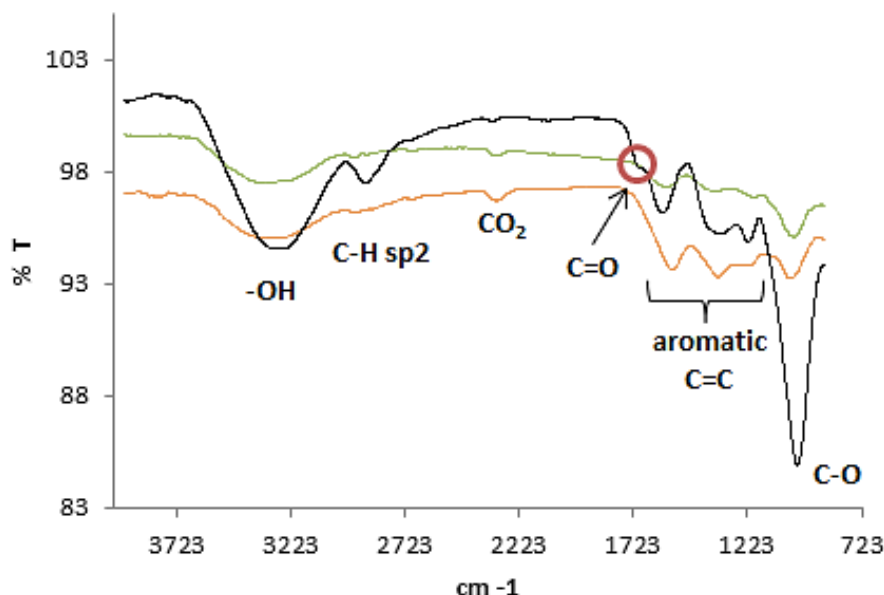


Fig. 2. Overlay FT-IR spectra of raw-PP, PPAC-K40 and PPAC-P20

Production of Graphite Oxide Catalyst (GO/AC)

GO has the tendency to stack together due to pi-to-pi interactions and enfoldment of graphite sheets. Its small interlamellar spacing prevents its utilization. Thus, structural defects from incorporating activated carbon can be useful for extending the functionalization of graphite [22].

Variation of impregnation temperature

Previous work of Sun et al. made use of Graphite Oxide (GO) with P-Phenylene Diamine (PPD) at temperature above and below room temperature ($10\text{--}90^\circ\text{C}$) to produce the desired modified composites. Results showed structural changes as seen on its XRD patterns with interlamellar spacing d values increasing as reaction temperature increased [23]. Thus, in this study, heat treatment was tested in the impregnation of AC and GO. The effect of applying heat during impregnation was assessed based on its FAME yield. For all reaction conditions, an increase in impregnation temperature was directly proportional to the product yield. It can be inferred from the results that at elevated temperature, efficacy of the impregnated GO as catalyst was enhanced. The composites obtained at different temperatures were denoted as, AC/GO-T ($T=30, 50$ and 90) in **Figure 3**, where T represents the temperature.

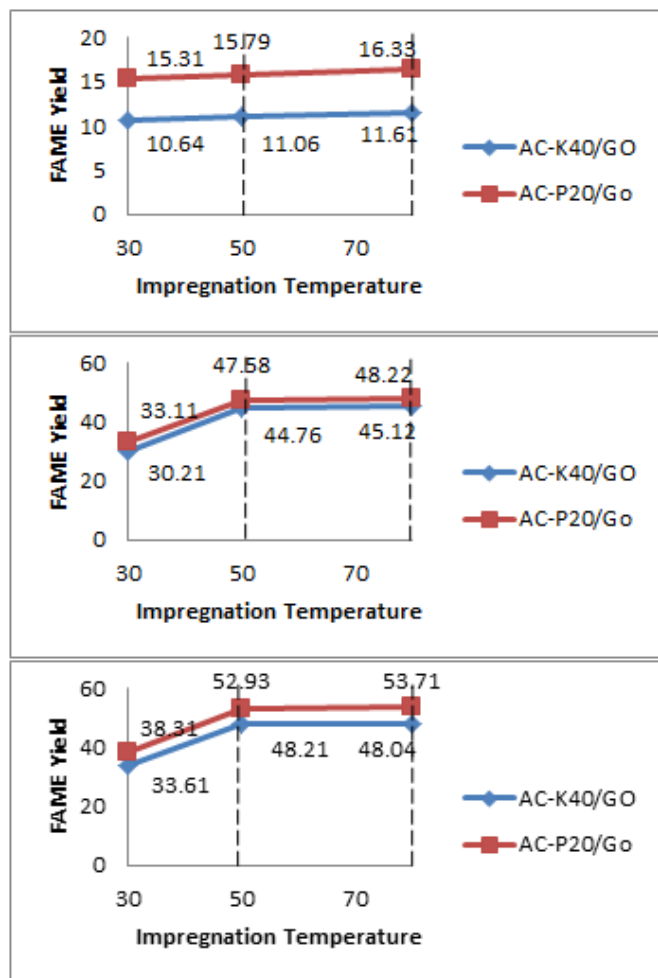


Fig. 3. FAME yield at different impregnation temperature for 1:6 (Top), 1:12 (Middle) and 1:18 (Bottom) oil to methanol molar ratio

Surface Morphology Analysis

SEM was used to investigate the impregnation of graphite oxide. A closer look of graphite oxide, as shown in its SEM micrograph in **Figure 4A**, shows a rough surface with debris that can be attributed to its oxygen functionalities [24]. PPAC/GO composites, at impregnation temperature of 80 °C, then showed a combination of porous structure and flat surfaces which can be an indication that PPAC and GO interacted [25]. The SEM images of PPAC-H₃PO₄/GO composites indicate the surface modification of GO after functionalization at different temperatures, as shown in **Figure 5**. At impregnation of RT, the surface contained several pits. This may mean partial adsorption of GO to the pores of AC but considering the nature of the material, it could be interpreted in another way. A fully anhydrous GO is very hard to achieve since GO consists of many ‘micro/nano’ pockets that can trap both H₂O and CO₂. Here, CO₂ may have freely chemisorbed onto the GO scaffold via G-OH to G-O-C(=O)-O-G causing interaction between CO₂ and H₂O. This phenomenon cannot be observed in AC since it has already undergone heating at extreme temperatures. However, the H₂O from GO may have reacted with the oxygen functionalities present in AC thereby stunting the progress in the impregnation of GO to AC. The temperature is most likely a key factor in the incomplete interaction of AC and GO as was also resulted in the study of Sharma et al. [26]. In PPAC-H₃PO₄/GO_{T50°C}, disorder in the composite surface is clearly apparent in its SEM image. This was probably due to the higher interaction between AC and GO, which caused GO molecules to react with the oxygen-containing groups in AC through polymerization [27]. Compared to PPAC-H₃PO₄/GO_{T80°C}, the GO surface appeared flatter and smoother which could mean complete impregnation of GO to AC.

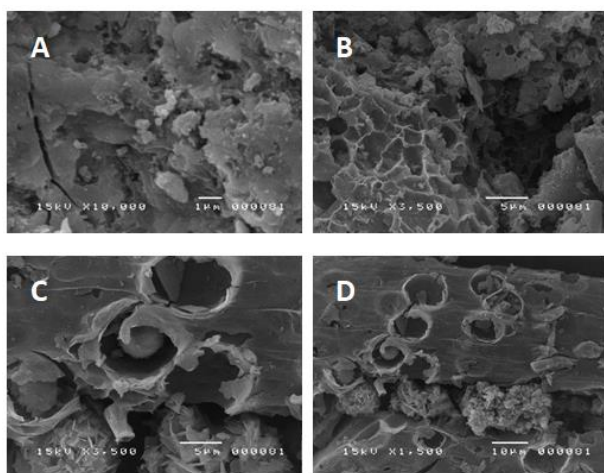


Fig. 4. SEM micrographs of GO (a), PPAC-KOH/GO_{T80°C} (b) and PPAC-H₃PO₄/GO_{T80°C} (c and d) at 10,000x, 3,500x, 3,500x and 1,500x, respectively

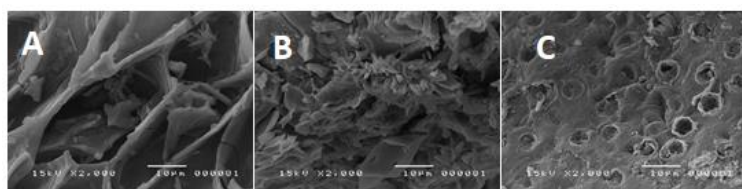


Fig. 5. SEM micrographs of PPAC-H₃PO₄/GO at RT (a), T50, (b) and T80 (c) at 2,000x magnification.

Surface Functional Group Analysis

The impregnation of AC/GO can be supported by the FT-IR peaks shown in **Figures 6 and 7**. The absence of absorption peaks caused by carboxyl group (O-C=O) and epoxy group (C-O-C) is an indication that only non-covalent hydrogen and very few ionic bonds occurred in the impregnation [28]. In both acid and base treated AC/GO composites, the O- H bond is most likely strengthened by a stronger interaction in non-covalent hydrogen bonding as seen in shifting of the band to a higher wavenumber. Unimpregnated GO and AC have a very low peak intensity when overlaid with AC/GO composites. The increase in peak intensity after impregnation could mean that the GO was indeed impregnated.

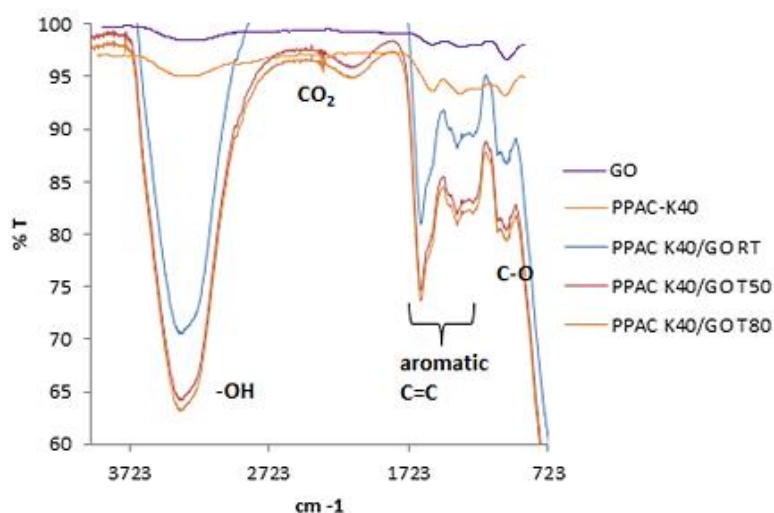


Fig. 6. Overlay FT-IR Spectra of KOH-treated AC/GO composites

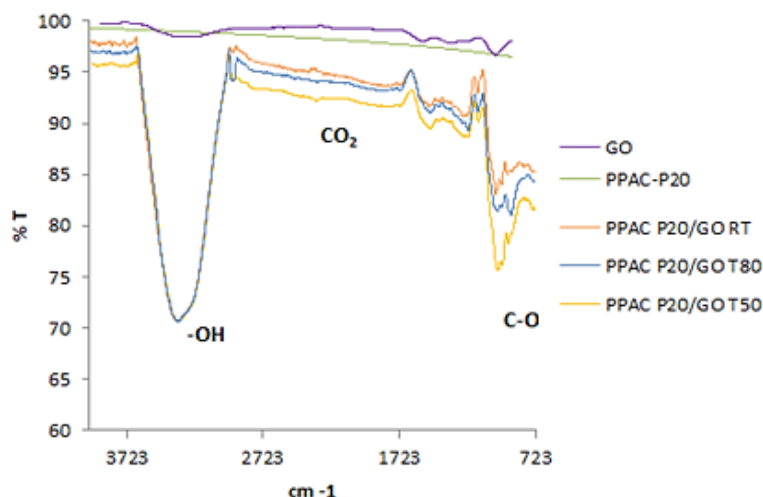


Fig. 7. Overlay FT-IR Spectra of H₃PO₄-treated AC/GO composites

Utilization of GO/AC in the Transesterification of Coconut Oil

The potential of GO-AC catalysts was investigated via transesterification reaction using different coconut oil to methanol molar ratio (1:6, 1:12 and 1:18). The reaction time, temperature and catalyst loading were held constant at 1.5 hours, 65°C, and 1.5 (w/w)%, respectively. A trend was observed in relation to the amount of methanol used to the FAME yield. As the oil to methanol molar ratio increases, FAME yield also increases. As shown in **Table 1**, a notable difference was observed between 1:6 and 1:12 molar ratio with slight deviation between 1:12 and 1:18 molar ratio. The % yield of Fatty acid methyl ester (FAME) of various transesterification reactions were investigated based on GC analysis. The 1:18 oil to methanol molar ratio exhibits the highest yield of FAME with an average value of 53.58 % ± 0.14. Another thing that can be observed is that the base-treated AC/GO composites exhibit lower catalytic activity than acid-treated AC/GO composites [29]. The extension of functionalization of GO is indeed affected by the species involved in the impregnation as shown in **Figure 8**. Thus, the expected extension of functionalization of GO was achieved because of the successful interaction of PPAC and GO during impregnation.

Table 1. FAME Yield of prepared catalysts at different coconut oil to methanol molar ratio

Catalyst		FAME Yield		
		At 1:6 oil to methanol ratio	At 1:12 oil to methanol ratio	At 1:18 oil to methanol ratio
A	Without catalyst	0.85	1.17	1.23
B	PPAC-KOH T _{400°C}	10.93	12.31	12.72
C	PPAC-H ₃ PO ₄ T _{800°C}	15.68	23.16	28.63
D	PPAC-KOH/GO _{RT}	10.64	30.21	33.61
E	PPAC-H ₃ PO ₄ /GO _{RT}	15.31	33.11	38.31
F	PPAC-KOH/GO _{T50°C}	11.06	44.76	48.21
G	PPAC-H ₃ PO ₄ /GO _{T50°C}	15.79	47.58	52.93
H	PPAC-KOH/GO _{T80°C}	11.61	45.12	48.04
I	PPAC-H ₃ PO ₄ /GO _{T80°C}	16.33	48.22	53.71

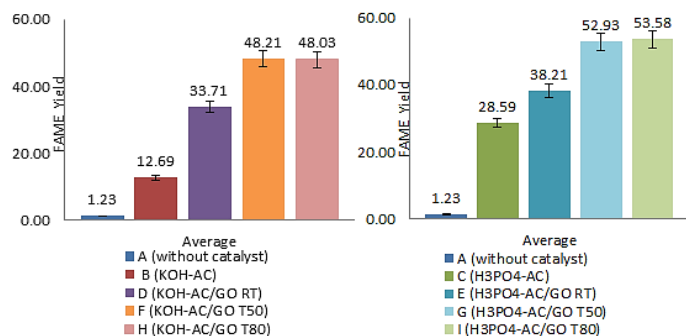


Fig. 8. Comparison of FAME Yield values at optimum oil to methanol ratio

CONCLUSIONS

The structural changes brought about by the impregnation of activated carbon from pineapple peel (PPAC) with graphite oxide (GO) were useful for extending the utilization and functionalization of graphite oxide as catalyst. The following conditions were found to be optimum to produce biodiesel: 1:18 oil to methanol molar ratio, 1.5% catalyst loading, 60 °C reaction temperature, and 90 min reaction time. PPAC-H₃PO₄/GO T80°C gave the highest yield of 53.71%.

DECLARATIONS

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

Ethics approval This article does not contain any studies with human participants or animals performed by any of the authors.

Consent to participate Informed consent is not applicable.

Informed consent Informed consent is not applicable.

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