FORMATION OF ALKYL-ESTER AS EFFECT OF IRRADIATION ^{60}Co GAMMA FROM OIL WASTE: PRELIMINARY STUDY AS CANDIDATE OF BIODIESEL

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Biodiesel is an alternative fuel produced from renewable vegetable or animal sources. This study aims to analyse the influence of cobalt-60 gamma irradiation on the yield results of alkyl ester as candidate of biodiesel from cooking oil waste. Biodiesel is synthesized from cooking oil through two main processes. The first step involved esterification using methanol in a 1:6 molar ratio and 0.05% acid catalyst (by weight of oil). The second step was transesterification reaction, which was carried out using methanol and NaOH as a catalyst, followed by exposure to gamma rays at doses ranging from 20 to 35 kGy. Fourier Transform Infrared Spectroscopy (FTIR) was employed to confirm the formation of ester functional groups characteristic of fatty acid methyl esters (FAME). The spectrum showed absorption bands at 1110 cm^{-1} (C–O stretching of esters) and 2980 cm^{-1} (C–H stretching of aliphatic chains). These findings indicate that gamma irradiation can enhance biodiesel production without additional chemical modification. As a preliminary study, the claim that irradiation accelerates transesterification must be supported by quantitative data on conversion (e.g. GC or ^{1}H-NMR).

Keywords: *biodiesel, cooking oil, waste, cobalt-60, gamma*

1. Introduction

The exponential growth of the world's population, combined with a high standard of living, has led to a sharp increase in energy consumption [1,2]. Despite the enormous demand, fossil fuel reserves have significantly declined and are now

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regarded as one of the primary contributors to global warming, evidenced by a rise in greenhouse gases in recent years. In the Paris Agreement, the key concession is to emphasize that to limit global warming to 1.5°C, greenhouse gas emissions must peak by 2025 at the latest and decrease by 43% by 2030 [3]. However, implementing the Paris Agreement involves maintaining the Earth's temperature and addressing energy needs; thus, alternative energy must be the primary focus at this time. One viable alternative is energy derived from renewable sources, such as plants or animals, commonly referred to as biodiesel, which is typically the main source of diesel derived from biomass at a specific concentration.

The advantages of using alternative fuels include improved emissions, biodegradability, and a reduced contribution to rising CO_2 levels in the atmosphere [4,19,20]. The raw materials for producing biodiesel typically come from vegetable oils. Commonly used vegetable oils include palm oil (cooking oil), castor oil, soybean oil, and Napoleonic oil (locally owned and operated business offering bulk fuel). Indonesia boasts the largest oil palm plantation in Southeast Asia. Cooking oil is often reused for frying multiple times, sometimes until it turns dark brown or black, before being discarded. This repeated use of cooking oil poses significant health risks. During its use, cooking oil undergoes chemical changes due to oxidation and hydrolysis, which can lead to its degradation. To address this issue, waste cooking oil can be repurposed as a raw material for biodiesel production [5]. Biodiesel serves as an environmentally friendly alternative fuel, with the benefit of reducing exhaust gas emissions, including hydrocarbons (HC), carbon monoxide (CO), sulfur oxides (SO), and other particulates [6,7].

The use of waste cooking oil, or used vegetable oil, as a feedstock for biodiesel production has garnered significant interest due to its availability, low cost, and the environmental benefits of repurposing a waste stream [8]. However, producing biodiesel from used cooking oil can be challenging because of the presence of contaminants and the need for effective catalyst systems. The process of making biodiesel involves an alcoholization reaction, which is an equilibrium reaction with minimal reaction heat. The alcohol used is a short-chain alcohol, such as methanol, ethanol, or buthanol. Methanol and ethanol can be easily produced from vegetable materials. Ethanol yields less ethyl ester and leaves behind a considerable amount of residual carbon. Methanol, in addition to being less expensive, is also the most commonly used alcohol. Catalysts are employed to accelerate the reaction process [9].

Methanol and ethanol are the types of alcohol that are widely used in industry, as these two types of alcohol provide a relatively faster reaction. The reaction with alcohol with a lower boiling point is carried out at a temperature of 70-85 ºC, while the reaction with alcohol with a high boiling point is carried out at a temperature of 200-250°C. The reactor used is operated in a dry state, and the level of free fatty acids present in the oil or fat must be small. The concentration of the catalyst will be reduced because water and free fatty acids will react with the catalyst, which is alkaline and form a soap.

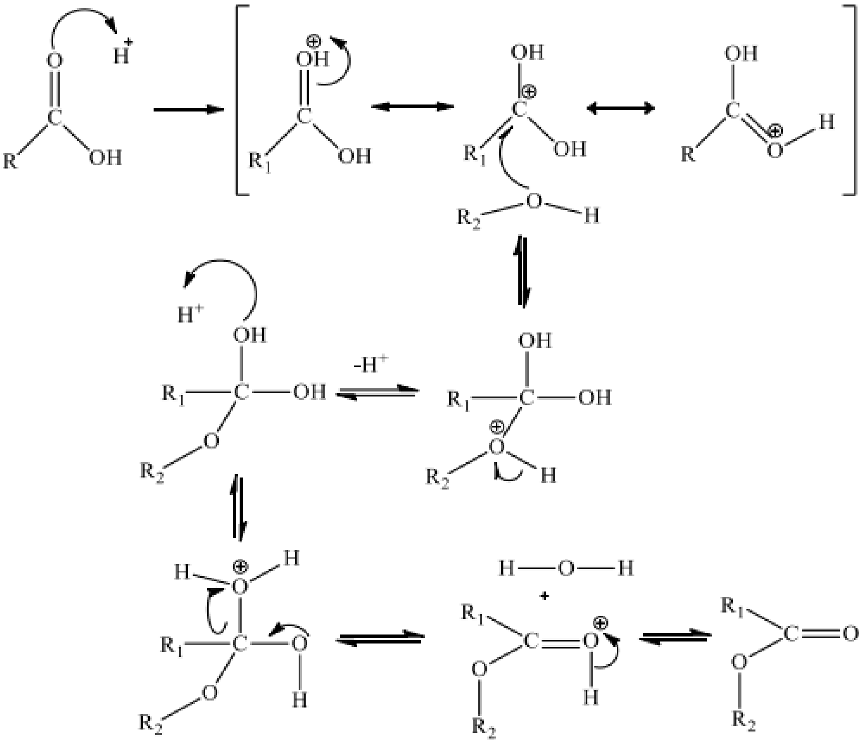

Fig. 1. Fatty acid esterification mechanism [9]

Fig. 1 showed fatty acid esterification mechanism from alkyl ester used cooking oil can be produced biodiesel (methyl ester). Transesterification is the replacement of the alcohol group of an ester with alcohol, so it is similar to the hydrolysis reaction, but not water to hydrolyze but alcohol, so it is called alcoholization. Transesterification is an equilibrium reaction to shift the reaction to the right, usually using short-action alcohols in excess or taking one of the mixed products; methanol is often used because it is cheaper, possibly with other alcohols such as ethanol. Commonly used catalysts are strong bases such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). This catalyst is mixed with alcohol (usually methanol) to form methoxide [10,11].

From *Fig.. 2*, transesterification reactions show that alcohol serves as a crucial acyl acceptor, allowing the conversion of esters by transferring the acyl group from a triglyceride or ester compound to the alcohol molecule. This reaction occurs in the presence of a catalyst, typically an acid or base, which aids in breaking the ester bond and forming a new ester bond between the acyl group and the alcohol.

The selection of alcohol, such as methanol or ethanol, significantly influences the reaction's efficiency, yield, and product properties due to its molecular structure and reactivity. For example, methanol is commonly used in biodiesel production because it enhances the reaction rate and conversion efficiency due to its polarity and low molecular weight. In this reaction, methanol accepts the acyl groups from the triglyceride's fatty acid chains, forming fatty acid methyl esters (FAME) as the primary biodiesel components, while glycerol is released as a byproduct. The role of alcohol as an acyl acceptor is crucial for transforming triglycerides into a more sustainable and usable form of biofuel.

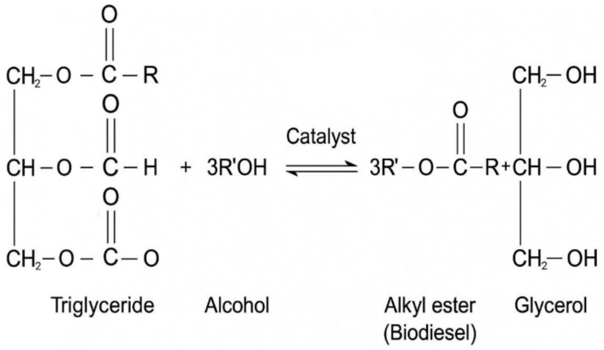

Fig. 2. Reaction of biodiesel production from ester and alcohol [12]

One promising approach to enhancing the biodiesel production process is the use of gamma irradiation from a Cobalt-60 (Co-60) source, Gamma rays from cobalt-60 are high-energy electromagnetic waves that can break chemical bonds or produce free radicals, which may enhance reaction rates or modify the structure of reactants in biodiesel synthesis. Gamma radiation has numerous advantages as an initiator, including the fact that it produces no residue, may be utilized at any temperature and in any application, and allows for controlled grafting or degradation [13]. In biodiesel production, gamma irradiation may assist in transesterification by generating reactive species or altering the viscosity and reactivity of the oil, leading to higher conversion. Additionally, the use of enzymatic catalysts, such as lipases, can provide advantages over traditional chemical catalysts, including milder reaction conditions and the ability to handle feedstocks with high free fatty acids [14,15,16].

2. Materials and Methods

2.1. Preparation of tools and materials

This study used various laboratory equipment to support the transesterification process of waste cooking oil into biodiesel. Chemical glassware, such as beaker glass (Pyrex), volumetric flasks (Pyrex), volume pipettes (Pyrex), Pasteur pipettes (Pyrex), etc, were utilized for mixing and preparing the materials. This research also used Analytical balances (Kern ABS 220-4N) for measuring. A distillation apparatus was employed to separate reaction products based on their boiling points, while a separating funnel was used to separate the liquid biodiesel phase from glycerol, a byproduct of the transesterification process. A stand was used to support glassware and funnels during the process to ensure stability and safety. Additionally, an irradiation container was used to ensure that all components were exposed to appropriate light during the reaction if irradiation was required in the preparation or reaction optimization stage.

Fig. 3. Tools and materials equipment

The raw material in the biodiesel production process was waste cooking oil as the triglyceride source as seen on Fig. 3. This oil was purified first to remove impurities that might inhibit the reaction. Sodium hydroxide (NaOH) was used as a base catalyst to accelerate the reaction rate. Methanol, serving as an alcohol agent or acyl acceptor, was mixed with the waste cooking oil and catalyst in the transesterification process to produce methyl esters (biodiesel) and glycerol. This combination of equipment and purified materials enabled an efficient transesterification reaction. Used cooking oil was obtained from used frying food at a restaurant in the Yogyakarta area, Indonesia. Used cooking oil was left to separate impurity by precipitation; then, the oil was filtered to separate pure cooking oil and impurities. Clear scheme of operations for the entire process can be showed on Fig. 4.

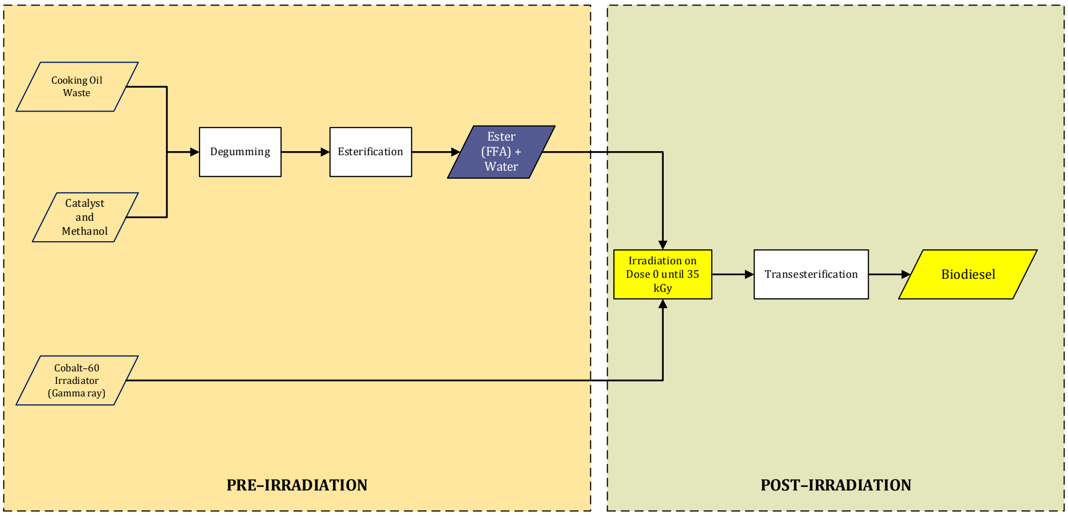

Fig. 4. Scheme of operations for the entire process

2.2. Degumming

150 mL of used cooking oil mixed with 0.5% concentrated HCl w/w and 20 mL of water with 0.5% NaOH w/w; the mixture was heated at 120°C using a distillation circuit.

2.3. Esterification

Degumming solution; 98% methanol p.a. by Merck (molar ratio 1:6); and 0.05% v/v p.a. H_2SO_4 as catalyst were mixed in 60°C for 60 min. The mixed solution was precipitated for 24 h and filtrate separated as esterification yield. The esterification step is carried out before transesterification, especially if the raw material used has a high free fatty acid (FFA), such as cooking oil waste. Esterification aims to reduce the FFA content and convert it into an ester through a reaction with methanol using an acid catalyst. The FFA content can be minimized by performing esterification first, allowing the transesterification process to proceed more efficiently and produce high-quality methyl esters. The esterified oil obtained from this step was subsequently subjected to irradiation before entering the transesterification process.

2.4. Irradiation using ^{60}Co

After completing the esterification step, the esterified oil was irradiated by ^{60}Co at the irradiation facility of the Polytechnic Institute of Nuclear Technology, Indonesia. The applied doses were 0 kGy, 5 kGy, 20 kGy, and 35 kGy. This irradiation step was positioned between esterification and transesterification to investigate its effect on improving molecular reactivity and enhancing the efficiency of the subsequent transesterification process.

2.5. Transesterification

After irradiation, 98% methanol with a molar ratio of 1:6 mixed with 1% w/w NaOH, then homogenized. The methanol-NaOH mixture was put in an alkyl ester solution (Biodiesel) to be stirred for 2 h at a temperature of 60°C, and then precipitated using a separation funnel for 24 h to be discarded from the raffinate.

2.6. Fourier Transform Infra Red (FTIR) Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to confirm the presence of ester functional groups as indicator of biodiesel. Spectra were obtained in the range of 4000–500 cm^{-1}, focusing on the identification of characteristic peaks of fatty acid methyl esters (FAME), such as C=O and C–O stretching.

2.7. Yield Measurement

The biodiesel yield was measured by calculating the volume of biodiesel obtained from a fixed initial volume of cooking oil (per batch). The yield was recorded in milliliters and compared across all irradiation dose levels.

3. Results and discussion

3.1. Yield Results

The primary research is measurement of biodiesel production outcomes. Irradiation can be used as alternative methods to convert waste cooking oil into biodiesel.

Table 1.

Comparation of yield type on various dose gamma irradiation

Yield Type	**Gamma Irradiation Dose**			
	0 kGy	5 kGy	20 kGy	35 kGy
Feed (mL)	150	150	150	150
Esterification (mL)	125.3	125.3	125.3	125.3
Alkyl Ester (Biodiesel) Yield (mL)	87.3	93.4	101.1	104.7
Mass Balance Ratio (regarding Biodiesel Yield) (%)	58.2	62.2	67.4	69.8

Table 1 showed that increasing dose gamma irradiation affected on biodiesel yield. Gamma irradiation can generate free radicals in vegetable oil that is used for producing biodiesel. These highly reactive free radicals can speed up main reaction of biodiesel formation.

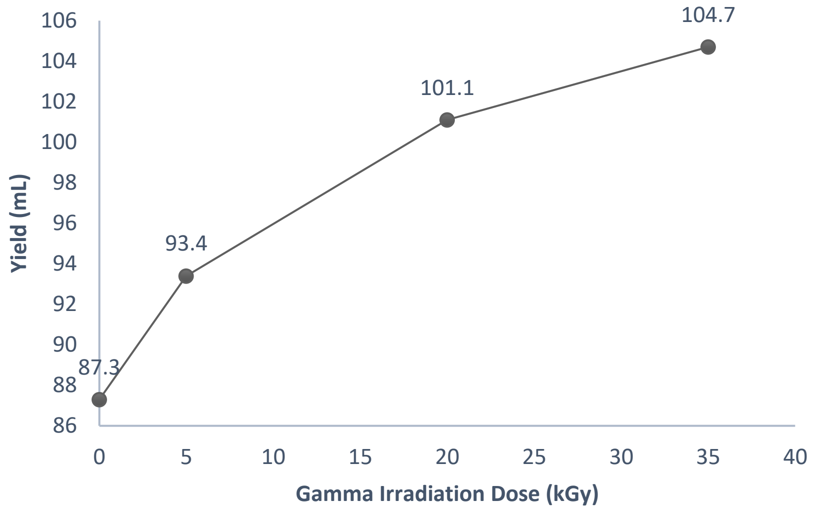

Fig. 5. Graph effect of gamma irradiation dose (kGy) on yield of biodiesel (mL)

As the dose of irradiation increases, the number of free radicals produced rises. Thus, the speed and efficiency of the transesterification process also increase. Breaking alkene (C-C) and ester (C-O) bonds can also facilitate transesterification reactions with methanol or ethanol as the molecules become more reactive. As a result, higher irradiation dose gamma affected on higher amount of broken bonds and volume biodiesel. From Fig. 5 the graph indicates a general increase in biodiesel volume as the irradiation dose increases from 20 kGy to 35 kGy. This trend suggests that gamma irradiation enhances the transesterification process, likely by promoting molecular agitation or the formation of reactive radicals that accelerate ester conversion.

The highest yield was recorded at 35 kGy, reaching a volume of 104.7 mL. This finding demonstrates the potential of gamma irradiation to improve biodiesel production efficiency. However, it is also important to consider the saturation point, beyond which further irradiation may not provide significant benefits or could even degrade the product.

III.2. Fourier Transform Infrared Spectroscopy (FTIR) Results

FTIR spectroscopy was employed to investigate changes in the functional groups of biodiesel compared with the original used cooking oil feedstock. Fig. 6 presents the FTIR spectra, and Table 2 summarizes the identified peaks and functional groups.

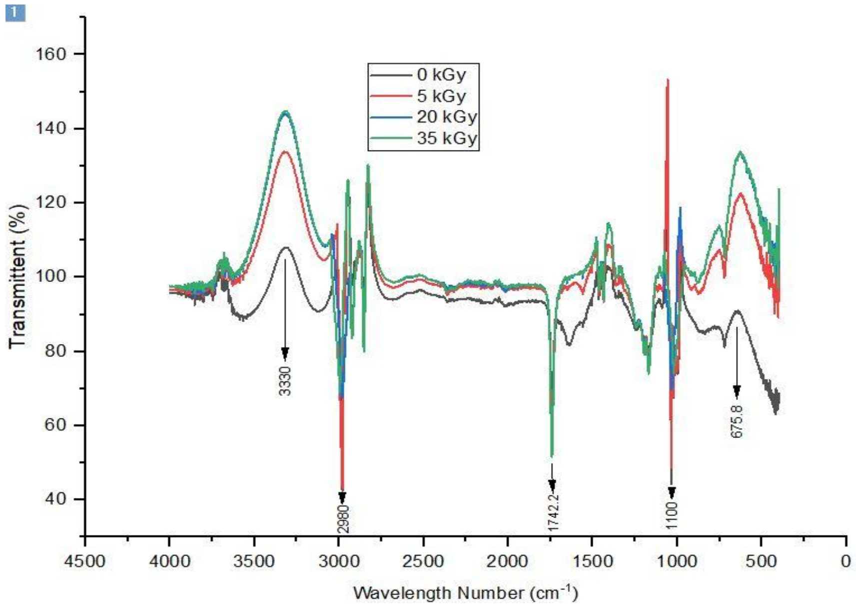

Fig. 6. FTIR spectrum of irradiated waste cooking oil on various dose gamma irradiation

The results of this FTIR graph are then analyzed to describe and shape the spectrum and determine the difference in the bonds between the guts and the names of the clusters produced on the spectrum.

Table 2.

Comparison of flame on non-irradiated and irradiated biodiesel results with dose variations

No	**Wavelength (cm^{-1})**	**Bonding**	**Function cluster name**	**Information**
1	1110-1150	C-O	Ester[1]	Howl
2	1740	C=O	Ester (FAME)[1]	Howl
3	2850-2980	C-H	Methylene[2]	Howl, symmetries
4	3200-3600	O-H	Alcohol[1]	Symmetries
5	1600	C=C	Alkene	Irregular
6	675.8	-CH=CH-	alkene	symmetries

Based on the spectral data in Fig. 6 and Table 2, characteristic absorption bands confirm the formation of methyl esters (biodiesel). A strong peak at 1740 cm^{-1} corresponds to the C=O stretching vibration of ester carbonyl groups, which is the main fingerprint of successful transesterification [18]. The 1110 cm^{-1} band indicates C–O stretching of ethers/esters, further confirming methyl ester formation. The 2980 cm^{-1} band (C–H stretching) and the 3330 cm^{-1} band (O–H stretching) represent alkyl chains and possible residual methanol or free fatty acids. In addition, bands at 1600 cm^{-1} (C=C stretching) and 675 cm^{-1} (CH=CH bending) confirm the presence of unsaturated fatty acid chains in biodiesel.

FTIR shows a band at ~1600 cm^{-1} consistent with C=C vibrations, indicating the presence of unsaturated carbon-carbon bonds in the fatty acid chain, the term 'alkenes' refers to compounds containing C=C (unsaturated) double bonds. Variations in irradiation doses can affect the stability of these bonds, with high doses potentially leading to bond breakage. The peak at wave number 3330 cm-1, which indicates the presence of O-H bond vibrations, indicates the presence of hydroxyl groups, which can be derived from residual methanol or from free fatty acids. Higher doses of irradiation can break these bonds, reducing peak intensity. The peak at the wave number of 675.8 cm^{-1} indicates the –CH=CH- bond vibration of the unsaturated fatty acid chain [21]. These peaks indicate the presence of symmetrical double bonds. Higher doses of irradiation can lead to isomerization or breakage of these bonds, which will alter the composition of the biodiesel.

As the output of the FTIR analysis of the biodiesel price, the relics of some functional groups reflect residual compounds from the transesterification and irradiation processes. C-O at wave number 1110 cm^{-1} indicates the group for ether, which can be synthesized from the additive reaction of layered compounds throughout the process of transesterification or irradiation. The O-H cluster at the wave number of 3330 cm^{-1} indicates alcohol and possibly residual methanol that has not yet reacted well. The introduction of the C=O group at the wave number 1742.2 cm^{-1} indicates the presence of esters as the main element of biodiesel. Of these five central FTIR spectral peaks, the intensity still shows that the compound remains after gamma up to a dose of 35 kGy. The irradiation effect is able to increase the efficiency of transesterification at times, symbolized by the greater basin intensity of the ester group, but not completely, giving no residual compounds such as methanol and glycerol. The C=C group at 1600 cm^{-1} reflects the non-permanent fatty acids that there is a C-O-C group with minus carbon not fully "passing" to the ester, and we are caught having another unsaturated carbon chain, i.e., the group –CH=CH– number 675.8 cm^{-1} [22].

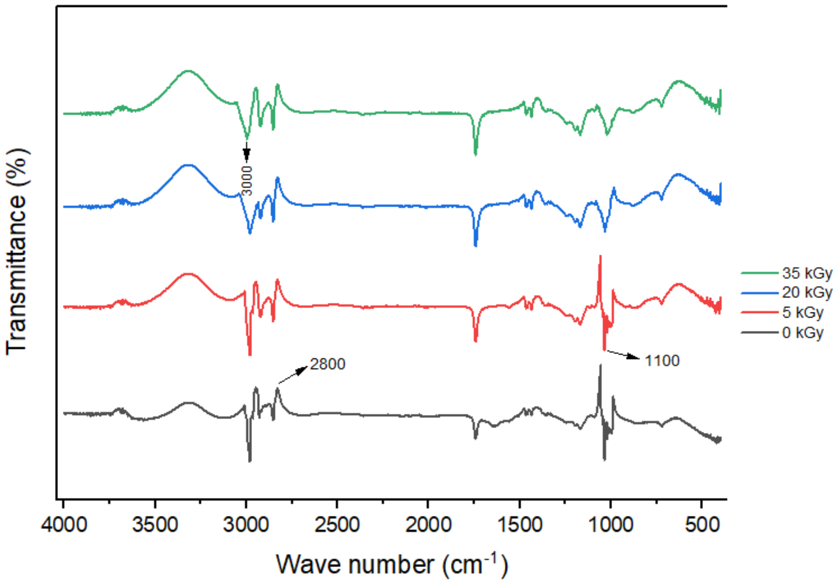

Fig. 7. FTIR spectrum of biodiesel formation by various dose gamma irradiation (using stacked line spectrum)

Gamma irradiation can break the C-O bond, resulting in a change in the FTIR spectrum that indicates a decrease or loss of peaks in this wave number. When biodiesel molecules (methyl esters) are exposed to gamma rays, the C-H, C=O, and C-O bonds can break, producing free radicals. These free radicals can initiate transesterification reactions more efficiently, accelerating the conversion of oils into methyl esters (biodiesel). Fig. 7 showed the FTIR spectrum for biodiesel samples showed a characteristic peak at ~1742 cm^{-1} indicating the C=O vibration of the ester (FAME), as well as a C–O peak in the range of ~1110–1150 cm^{-1} consistent with the C–O ester vibration. Wide bands at 3200–3600 cm^{-1} indicate the presence of O–H (possible water or alcohol residue). At irradiation doses of 20 kGy and 35 kGy, changes in the FTIR spectrum will be more pronounced. The peaks for functional groups such as C-O, C=O, C-H, and O-H show significant changes in intensity and slight shifts in position. Gamma irradiation can cause the formation of radicals that have the potential to affect molecular structure and reactivity. However, the claim that irradiation *accelerates transesterification* must be supported by quantitative data on conversion (e.g. GC or ^{1}H-NMR) [23].

Based on this study data, the optimal irradiation dose to yield 35 kGy was concluded (biodiesel yield and quality parameters were ameliorated up to this irradiation dose), as shown by the findings from this work. But, the in-depth examination at higher doses is missing, which portends questions about further increases in the dose of irradiation. Even higher doses would likely cause the transesterification reaction to become even better since they would create more free radicals, which would be good for yield and quality. On the other hand, too much irradiation would also degrade molecular building blocks excessively, which may lead to adverse byproducts or degrade biodiesel quality. In future studies, irradiation doses should be set beyond 35 kGy to fully investigate the threshold beyond which the benefits of irradiation are never more than acceptable without damaging biodiesel quality and delivering more insight into the whole process.

4. Conclusion

From the results of this study, it can be concluded that increasing the dose of cobalt-60 gamma irradiation positively influences the biodiesel yield from waste cooking oil, with the highest volume of 104.7 mL obtained at a dose of 35 kGy. The O-H cluster at the wave number of 3330 cm^{-1} indicates alcohol and possibly residual methanol that has not yet reacted well. The introduction of the C=O group at the wave number 1742.2 cm^{-1} indicates the presence of esters as the main element of biodiesel. Of these five central FTIR spectral peaks, the intensity still shows that the compound remains after gamma up to a dose of 35 kGy. These findings indicate that gamma irradiation can enhance biodiesel production without additional chemical modification. As a preliminary study, the claim that irradiation *accelerates transesterification* must be supported by quantitative data on conversion (e.g. GC or ^{1}H-NMR)

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