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APPLICATION METHODS LIQUID SCINTILLATION COUNTING (LSC) IN DETERMINING CONCENTRATION OF BIO SOLAR

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ABSTRACT

Research on the use of a liquid scintillation counter method in determining the concentration of biodiesel in the market has been carried out by taking samples of biodiesel in Pertamina. Preparation done by creating a standard biodiesel and biodiesel mixed with diesel at a certain concentration. Biodiesel is chemically formed by the transesterification and esterification process is converted into methyl ester triglycerides with the help of H_2SO_4 and NaOH as a catalyst. Total carbon in the sample solution is 1.5690 g/ 8 mL obtained through methods spekktrofotometri uv-vis. The method of determining the concentration of a specific activity based on measurements of samples obtained from the shredded Liquid Scintilation Counter (LSC) Hidex 300 SL, ie 11.5902 DPM / gC. By using the specific activity of the sample and the specific activity was added to standard linear equations of the obtained sample concentration of biodiesel amounted to 18.81%.

Keywords: Biosolar, carbon-14, specific activity , LSC, UV-Vis

1. INTRODUCTION

Presidential Decree No. 5 of 2006 which states on national energy policy and a presidential instruction No. 1 of 2006 on the supply and use of biofuels as other fuels. The main consideration is the depletion of oil reserves in the bowels of the earth Indonesia and air pollution is a growing concern. This awareness is urged to use alternative renewable energy and cause less pollution [7].

Biosolar is one type of liquid fuel used in the combustion process in a motor vehicle. Biodiesel fuel sold in the market is a mixture of a number of products obtained from various processes. Through the process of mixing (blending) that the nature of the fuel can be set to provide the desired operating characteristics such as [8]. The obstacles faced by small and medium industries is how to perform quality assurance as a controller with accurate product quality, but at a low cost. Therefore, it is necessary to develop a quality test that can maintain the quality of biodiesel produced, making it safe for the user, with a short analysis times [9].

Member countries of the European Union to develop implementation methods Liquid scintilltion counting (LSC) directly to determine the quality based on the number of vegetable materials contained in biofuels. Liquid scintillation counting (LSC) method is a suitable method for measuring the quantity of biofuels through the determination of ^{14}C [4]. As is known in the petroleum activity ^{14}C fossils are very much different than the ^{14}C activity in vegetable materials. In the example of petroleum fossil ^{14}C activity is very small and even non-existent, while the vegetable material near the maximum is 15.3 DPM / gC [3]. A large number of vegetable ingredients that have been added in the petroleum fossil calculated based on the

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greater the activity of ^{14}C. The greater the ^{14}C activity tracked, the greater the proportion of vegetable material within the sample. Kristof and Logar (2013) has made measurements of biocomponents in fuels using bioethanol LSC them in ethanol, ethanol in gasoline, diesel fuel and HVO, HVO in gasoline.

2. METHOD

Materials Research

Research materials are palm oil, methanol p.a, H_2SO_4, NaOH p.a, Methyl Orange (MO), Phenolpthalein (PP), scintillator Ultima Gold and aquades.

Research Tools

The tools used in this study include tool rod stirrer, a bottle of reagent, funnel, separating funnel, erlenmeyer, beaker, Hot Plate / Heating, Klamp & stative, Balance, pycnometer, Pipette Volume, Spatula, Stirer, Thermometer, LSC Hidex 300 SL.

Determination of Total Carbon in The Sample [1]

Total carbon in the sample can be calculated from the organic carbon concentration obtained from the absorbance is measured using a UV-Vis spectrophotometer. Samples Biosolar, pipette of 0.5 ml is then added $K_2Cr_2O_7$ much as 5 mL and 7.5 mL of concentrated H_2SO_4. Then homogenized in let stand for 30 minutes. Furthermore, in preparation for testing, a standard solution of organic concentration of 0 ppm, 50 ppm, 100 ppm, 150 ppm, 200 ppm and 250 ppm were made with pipette 0 mL, 1 mL, 2 mL, 3 mL, 4 mL, 5 mL, from the mother liquor organic C 5000 mg / L. Each was added to 100 mL Nessler tube. Then added $K_2Cr_2O_7$ as much as 5 mL 1N. Allowed to stand for 24 hours. then each of the absorption solution is measured by a spectrophotometer wavelength of 561 nm.

Measurement of ^{14}C Activity in The Sample[6]

^{14}C activity in the samples and standards expressed in units of activity, which is the decay every minute (DPM) of carbon-14. The results of a sample census with a liquid scintillation counter Hidex 300 SL generate data in units of counts per minute (CPM) and the counter efficiency (TDCR) or E.

$$E = \frac{Cpm}{Dpm} \times 100\%$$

Determination of ^{14}C activity in the sample can be known through the enumeration of samples with LSC Hidex 300 SL. A homogeneous mixture of 8 mL of sample and 12 mL scintillator into 20 mL vial enumerated in this sample that has been marketed biodiesel and biodiesel (standard) with the LSC Hidex 300 SL at the time of enumeration 5-240 minutes.

3. RESULTS AND DISCUSSION

Determination of total carbon can be achieved by uv-vis spectrophotometer. Measurements were taken at each solution at a wavelength of 561 nm so that the resulting absorbance of each solution. Equation relationship between absorbance and concentration are directly proportional, so that the carbon concentration in each solution can be calculated. The results of measurements of total carbon in biodiesel is as follows:

Table 1. Total Carbon in The Sample

No	Sample	Total Carbon (g)
1	Biosolar 5%	3,1311
2	Biosolar 10%	2,8616
3	Biosolar 15%	2,7724
4	Biosolar 20%	2,9228
5	Biosolar 25%	3,1727
6	Biosolar X	1,5688

Total carbon was used to calculate the specific activity of ^{14}C which is expressed in units of disintegration per minute per unit mass of carbon (DPM / gC).

A sample census carried out in two phases, namely, the step of determining the optimum time of enumeration and the step of determining the average value of the sample count values at the optimum time. The following data is the result of the determination of the optimum time of enumeration of ^{14}C activity contained in the sample.

Table 2. Results of Enumeration Data for Optimum Timing Biosolar Shredded samples in a span of chopped 5-240 minutes.

		Sample		
No	Shredded Time (Minutes)	CPM	DPM	TDCR
1	5	1160,930	42638,780	0,027
2	10	469,050	6983,710	0,067
3	15	332,820	3296,580	0,100
4	30	220,040	1551,840	0,141
5	60	119,800	459,270	0,260
6	90	93,980	283,980	0,331
7	120	87,110	239,870	0,363
8	150	76,110	177,340	0,429
9	180	65,510	131,910	0,496
10	210	66,600	135,980	0,489
11	240	65,250	132,940	0,492

Information :
CPM = Value of chopped samples per minute
DPM = number of samples per minute decay
TDCR = Efficiency enumeration Sample

According to Table 4 it appears that at the time of counts during ranging from 5-180 minutes decreased from 1160.930 to 65.510 and the count values in the count values up to 180 minutes to 240 minutes of ^{14}C activity values ranging achieve stability. The cause is physical and chemical conditions of the sample solution with the scintillator is stabilized. Solution phase stability affect the efficiency of

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enumeration (TDCR) where efficiency enumeration ^{14}C high of approximately 80% or 0.8. Quenching effect causes the number of photons generated in the process pengemisian beta particles by the scintillator is reduced, causing a small enumeration efficiency. Blackouts or quenching source of oxygen or impurities in the bottle / vial is dissolved in the sample [2].

The timing of the enumeration conducted to determine the optimum time resulting value of DPM and the efficiency of enumeration (TDCR) stable as a sign that the process is running maximum sample census. The results of the count at the optimum time is used to calculate the specific activity of ^{14}C in the sample. The following data on the results of a sample census enumeration optimum time for 180 minutes with 5 repetitions.

Table 3. The value of average activity biodiesel sample at the time of enumeration optimum for 180 minutes

		Biosolar	**Samples**	
No.	**Shredded time (minutes)**	**CPM**	**DPM**	**TDCR**
1	180	67,790	144,880	0,467
2	180	69,390	150,800	0,460
3	180	62,230	121,490	0,512
4	180	62,030	116,690	0,513
5	180	65,810	120,531	0,546
	Average	**65,450**	**130,878**	**0,500**

Based on the above table, the value of the average CPM of 65.450 samples, the average value of DPM at 130.878 and average value TDCR is 0.500. The same treatment was also carried out against the background enumeration and standards by adding 12 ml of scintillator was added 8 mL vial containing the standard solution then enumerated with LSC Hidex 300 SL.

Determination of Specific Activity Sample and Standards

The specific activity of biodiesel and standard samples can be determined from the difference between the results of the count Disintegration Per Minute (DPM) sample of the results of the count Disintegration Per Minute (DPM) divided by the background levels of total carbon. The specific activity average (As) sample from the calculation of disintegrations per minute (DPM) per unit carbon future samples.

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Table 4. Data Specific Activities average ^{14}C Standard Solution

Standard	CPM	DPMs	DPMb	DPMk	g C	Specific Activity ^{14}C
5%	51,892	119,326	112,693	6,633	3,131	2.1184
10%	57,756	127,568	112,693	14,875	2,862	5.1974
15%	66,540	144,258	112,693	31,565	2,772	11.3871
20%	60,212	148,948	112,693	36,255	2,923	12.4033
25%	62,754	159,004	112,693	46,311	3,173	14.5953

Information :
DPMS = disintegration per minute standard
DPMB = disintegration per minute background
DPMk = DPMs - DPMb (Correction)
Efficiency = DPMs / CPM × 100

Based on data from a standard enumeration results in Table 4, shown specific activity of ^{14}C at the standard 5%, 10%, 15%, 20%, 25% respectively are 2.1184, 5.1974, 11.3871, 12.4033, and 14.5953. Graph of the relationship between the concentrations of the specific activity of the standard can be seen in Figure 1.

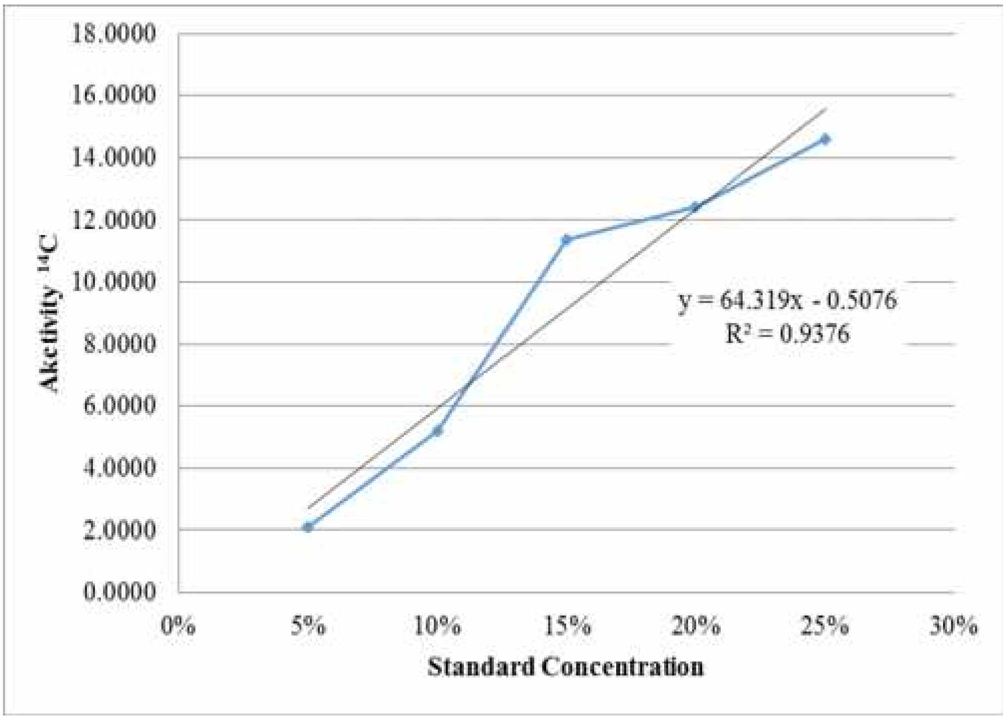

Figure 1. Graph of the relationship with the standard concentration of a specific activity

The value of a specific disintegration ^{14}C obtained showed that the number of atoms of ^{14}C decays per minute (DPM) in every single gram of carbon elements. The value of a specific disintegration (specific activity) obtained from the sample was 0.5460.

Table 5. Data Activities Specific average ^{14}C Bio Solar

	CPM	DPMs	DPMb	DPMk	g C	Specific Activity (DPM/gC)
Sample	65,450	130,878	112,693	18,185	1,569	11.5902

The concentration of the samples was determined from linear equations based on Figure 1. The concentration of the sample is obtained by 18.81%. This indicates that the sample contains a component of biodiesel.

4. CONCLUSION

Based on the research that has been done a great obtained specific activity of ^{14}C in samples of biodiesel based on measurements of ^{14}C activity through methods Liquid Scintillation Counting is 11.5902 DPM / gC and concentration of biodiesel is 18.81%.

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