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Optimization of the direct LSC method for determination of biogenic component in liquids by applying ^{14}C

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Abstract
Determination of fraction of biogenic component in liquid fuels by a direct radiocarbon measurement in liquid scintillation counter (direct-LSC method) has been validated by participation in the international intercomparison exercise. All the results for samples with the standard quench parameter SQP(E) value above ≈ 650 were accepted. Highly quenched sample of used edible oil was diluted with the ^{14}C-free petroleum ether to optimize the region of applicability. It was established that quantitative results were obtained for SQP(E) values above 700, while in the SQP(E) region between 700 and 600 only the qualitative results of f_{bio} can be taken.

Keywords Biogenic component · Liquid fuels · Radiocarbon method · Direct liquid scintillation counting · Optimization · Intercomparison

Introduction

A target of the European Union for decarbonisation and use of renewable energy sources is to cut carbon emissions by at least 40 % below 1990 levels by 2030 [1]. Renewable sources include various biofuels and advanced biofuels, as well as fuels from non-biogenic materials/sources. A promising alternative is co-processing of biogenic and petroleum derived liquids in the fluid catalytic cracker (FCC) in commercial oil refineries [2–4]. Production of various components of biogenic origin and their admixture with fossil fuels and incentives for use of bio-components initiated development of methods for accurate and precise quantification of the fraction of biogenic component in liquid fuels.

Radiocarbon methods based on various concentration of radiocarbon (^{14}C) in biogenic and fossil fuels has been proved to be one of the most accurate and reliable methods of biogenic fraction determination and the only method that directly quantifies the carbon of recent biological origin in a material [5–7]. The ^{14}C method can be successfully applied for determination of biogenic component in any type of samples and by different measurement techniques, such as accelerator mass spectrometry (AMS), liquid scintillation counting with benzene synthesis (LSC-B), liquid scintillation counting with absorption of CO_2 (LSC-A) [3, 5, 7–13]. However, these classical radiocarbon techniques, used for dating purposes mainly, can be expensive and time consuming although applicable to all kinds of material (solid, liquid, gaseous) [3, 5, 7].

When liquid fuels are concerned, an appropriate technique for determination of biogenic component fraction may be a so-called direct method of ^{14}C activity measurement in liquid by liquid scintillation counting ("direct LSC" in further text) [13–20]. The method is regarded as fast, simple, accurate and sensitive determination procedure for assessment of biogenic fraction in biofuels. The main problem of the method is related to different quenching properties of fuel mixtures, which basically consist of the fossil matrix (diesel or petrol) and various biogenic blends. Although the main idea of measurement is the same, there are some variations in calibration methods applied in different laboratories. Some calibration curves can function only for specific biocomponents in the single fossil fuels matrix. Other radiocarbon dating techniques, such as AMS [3, 8–10], LSC-B or LSC-A [5, 13] do not exhibit quenching problem, but they are more complicated since they require sample preparation, and are therefore more expensive and more time-consuming, as well as potentially dangerous if relatively large amounts of volatile fuels have to be combusted [13].

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At the Ruđer Bošković Institute (RBI) we have been implementing our own data evaluation method based on the quench properties of fuels (SQP(E)—Standard Quench Parameter in LSC Quantulus 1220) [13]. Similar approach of using a quench parameter curve has been applied earlier [16]. They used a quenching curve installed into the LSC instrument to determine count rates of the non-quenched sample, while in our method the quenching curves for fossil and modern samples have been prepared in the laboratory [13].

We participated in the international intercomparison study ILC/2018 *Content of biocomponent in liquid fuel samples*, which was organized in 2018 by the Institute of Ceramics and Building Materials (Opole, Poland) with the aim to validate our approach in data evaluation. We also used a sample of used edible oil, which has a potential of being used in FCC co-processing, to further optimize the direct LSC method.

Theory

Radiocarbon method for determination of biogenic component fraction in a sample is based on various concentrations of ^{14}C in biogenic material and in fossil fuels. Biogenic component reflects the ^{14}C activity of the contemporary atmospheric CO_2, and the fossil component is free of ^{14}C [5–7, 10, 13, 21–23].

A material can be composed of a biogenic component with fraction f_{bio} and a fossil component with fraction f_f

$$f_{bio} + f_f = 1. \quad (1)$$

The measured relative specific ^{14}C activity $a^{14}C_{mix}$ (in further text, the term "^{14}C activity" is used for $a^{14}C$ expressed in pMC—percent Modern Carbon) [11, 24] of such a mixed material can be presented as a combination of the fossil and biogenic components with their respective fractions f_f and f_{bio} and their respective ^{14}C activities $a^{14}C_f$ and $a^{14}C_{bio}$

$$a^{14}C_{mix} = f_f\, a^{14}C_f + f_{bio}\, a^{14}C_{bio}. \quad (2)$$

The $a^{14}C_f$ of fossil component is equal to 0 pMC since all the radiocarbon has been decayed in such a material. Therefore, the fraction of biogenic component can be found from the measured $a^{14}C_{mix}$ of a sample as [6, 10, 13, 16]

$$f_{bio} = a^{14}C_{mix}/a^{14}C_{bio} \quad (3)$$

The question remains what is the value of $a^{14}C_{bio}$ in (3) since the proper assignment of this value is critical for obtaining accurate results [9]. The $a^{14}C_{bio}$ refers to the biogenic ^{14}C activity from the year when the plant grew reflecting the ^{14}C content of atmospheric CO_2 during biomass growth. Before anthropogenic activities disturbed the natural distribution of ^{14}C in the atmosphere and biosphere during the twentieth century, the value of $a^{14}C_{bio}$ was 100 pMC [11, 25]. The $a^{14}C_{bio}$ has been changing in the second half of the twentieth century approaching almost twice the natural $a^{14}C_{bio}$ value in the early 1960s due to atmospheric bomb tests and declining since then [25]. The ASTM D6866 standard test method, issue 2012 [21] recommended the use of 105 pMC for contemporary biogenic material, and the issue from 2016 recommended 102 pMC [22]. However, the ASTM D6866-21 issue [23] recommends using the value of 100 pMC for biomass produced in recent years. This is corroborated by recent analysis of global atmospheric $a^{14}C$ [25]. Their data showed the average $a^{14}C$ in the atmosphere in the 2015–2019 period of 101.4 ± 0.5 pMC, with a decreasing trend towards 100 pMC. In addition, our own measurements of monthly atmospheric ^{14}C activities in the area of the City of Zagreb resulted in the average of $a^{14}C_{bio} = 100.0 \pm 2.2$ pMC for the period 2016–2020 [26].

Experimental

Interlaboratory comparison ILC/2018 *Content of biocomponent in liquid fuel samples* was organized by the Institute of Ceramics and Building Materials (Opole, Poland) in 2018. Seven liquid samples of diesel-based liquid fuels with unknown concentration of the biogenic component were distributed. Samples were of different colours (Fig. 1), from almost colourless sample LL/18/1267 (G) to dark yellow LL/18/0807 (C). Therefore, it was expected that they will show various quenching properties and various SQP(E) values.

Sample preparation consisted simply of mixing 10 ml of Ultima Gold F scintillation cocktail with 10 ml of liquid sample in low-potassium glass vials. The Ultra Low-Level Liquid Scintillation Spectrometer Quantulus 1220 (Wallac Oy, PerkinElmer) was used for measurement of ^{14}C activity. A quench indicating parameter in LSC Quantulus 1220 is the Spectral Quench Parameter of the External Standard,

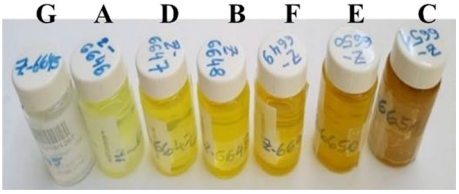

Fig. 1 Samples for the ILC/2018 Interlaboratory comparison *Content of biocomponent in liquid fuel samples*. Z-number is the laboratory identification code. Letters **A–G** refer to sample names in the following figures and in Table 1

SQP(E). The SQP(E) represents channel number of the 99th percentile of spectrum generated by external ^{152}Eu standard stored in Quantulus. Samples with higher quench level have lower SQP(E) values, which is a consequence of spectra shifting towards lower channels in the presence of a quenching agent. SQP(E) values were measured for each sample in all repetitions and the average value was determined. Spectra were acquired by WinQ software and evaluated by EasyView software. The Region of Interest (ROI) in the measured spectrum was between channels 145 and 570.

The RBI laboratory developed a data evaluation technique based on two calibration curves, the Background Calibration Curve (BCC) and the Modern Calibration Curve (MCC) (Fig. 1 in [13]). The BCC and MCC curves show the relation between the count rates and the values of the SQP(E) parameter for various ^{14}C-free liquids and pure biogenic liquids, respectively. As ^{14}C-free samples we used several types of pure fossil fuels, petroleum ether, commercial benzene and benzene (C_6H_6) used as ^{14}C-free background for ^{14}C dating synthetized in our laboratory. Various types of domestic oils (vegetable, sunflower, olive, pumpkin, flax, peanut, corn sprouts), bioethanol and benzene prepared from modern samples were used for MCC construction [13].

The procedure for the unknown sample consisted of: (1) measurement of the count rate (c_{sample}) and the SQP(E) value of a sample, (2) calculation of background (c_B) and modern (c_{bio}) count rates corresponding to the measured SQP(E) value of a sample based on the BCC and MCC curves, respectively, and (3) the fraction of the biogenic component in the liquid, f_{bio}, was calculated as the ratio of net count rates of the unknown sample and the modern net count rate at the same SQP(E) values.

$$f_{bio} = \left(c_{sample} - c_B\right) / \left(c_{bio} - c_B\right). \quad (4)$$

It was observed that the count rate of biogenic samples was indistinguishable from the background count rate at SQP(E) values below approximately 600 [13], thus resulting in the preliminary establishment of the applicability of the method for SQP(E) > 600. It was also shown that the method depended neither on the fossil matrix type nor on the biogenic additive [13].

^{14}C activity of three samples (A, C and E) from the ILC set was also measured by the AMS technique [27, 28], with a sample preparation adjusted to liquid fuels. A quartz tube with 4–7 mg of a sample, 500 mg of copper(II)-oxide and 100 mg of silver was put into the liquid nitrogen on a vacuum line and then evacuated to the pressure of 10^{-4} mbar and torch sealed. The sample was then combusted in a furnace at 850 °C for 8 h. Reduction of the obtained CO_2 to graphite was performed with Zn and Fe as a catalyst. Graphite was pressed on an Al target and the ^{14}C activity was measured at the accelerator at the Center for Applied Isotope Studies University of Georgia (USA) [29].

Results and discussion

Interlaboratory comparison samples

Table 1 shows measured f_{bio} values in seven ILC samples by the direct-LSC method and by applying BCC and MCC curves. As expected, the highest SQP(E) value was obtained for almost transparent colourless samples G, and the lowest for the darkest sample C. The SQP(E) value of 581 for sample C was lower than previously defined limit of the method applicability, SQP(E) = 600 [13], so the f_{bio} result for sample C was not reported.

The results of participation in the ILC were evaluated by the E_n value as:

$$E_n = \frac{f_{bio} - f_{bio-ILC}}{\sqrt{u_{RBI}^2 + u_{ILC}^2}}, \quad (5)$$

where f_{bio} and $f_{bio\text{-}ILC}$ are the biogenic fractions determined in the laboratory and the reference ILC value, respectively,

Table 1 Results of determination of biogenic component fraction in ILC samples: $f_{bio\text{-}ILC}$ is the reference value given by the organizer, f_{bio} is the value determined by the direct LSC method, AMS $a^{14}C$ is the ^{14}C activity determined by the AMS technique and is numerically equal to the fraction of biogenic component when the $a^{14}C_{bio}$ value of 100 pMC is used [23], and $\delta^{13}C$ is the relative abundance of ^{13}C in samples

Sample code	Sample name	SQP(E)	$f_{bio\text{-}ILC}$ (%)	f_{bio} (%)	AMS $a^{14}C$ (pMC)	$\delta^{13}C$ (‰)
Z-6646	A LL/18/0805	806	0.0 ± 0.9	0.34 ± 0.25	0.09 ± 0.01	–30.0
Z-6648	B LL/18/0806	724	7.0 ± 1.7	7.23 ± 0.60	–	–
Z-6651	C LL/18/0807	581	100 ± 2	–	97.21 ± 0.25	–31.0
Z-6647	D LL/18/1264	758	3.5 ± 1.1	4.44 ± 0.43	–	–
Z-6650	E LL/18/1265	609	30 ± 4	19.9 ± 2.4	28.6 ± 0.1	–30.4
Z-6649	F LL/18/1266	651	21 ± 3	18.4 ± 1.4	–	–
Z-6645	G LL/18/1267	875	7.6 ± 1.1	6.64 ± 0.30	–	–

and the u values are the corresponding uncertainties. Results were acceptable if $|E_n|<1$. The LSC f_{bio} results were acceptable for five samples, while the result for sample E was not acceptable (Fig. 2). This sample gave the SQP(E) value of 609 that was close to the limit of the method [13]. However, the f_{bio} value obtained for sample E indicated a significant fraction of the biogenic component and pointed to the possibility of existence of a region of limited applicability, or qualitative applicability, for which the upper limit remained to be determined.

All three AMS results were acceptable (Fig. 2) with $|E_n|<1$. The AMS results do not depend on the sample colour since the sample is first combusted and then graphite was prepared, as explained earlier. After combustion, a portion of obtained CO_2 was separated for the $\delta^{13}C$ determination by the isotope ratio mass spectrometry (IRMS) technique [28]. The obtained $\delta^{13}C$ values between –30 and –31 ‰ are typical values for fuels [3, 10] and also indicate absence of isotope fractionation during the sample preparation process.

Optimization by used edible oil sample

A sample of used edible oil (UEO) was used to further test the limits of applicability of the direct LSC method. Such sample types have been candidates for FCC co-processing [2]. The UEO sample (laboratory code Z-7226) was a dark sample giving the SQP(E) value of 546 (below the limit of the direct-LSC method applicability). The AMS measurement revealed $a^{14}C = 102.79 \pm 0.27$ pMC, i.e., $f_{bio} = 102.8 \pm 0.3$ % by applying the modern value of 100 pMC [23]. The $\delta^{13}C$ value was –29.6 ‰, as expected for the samples of biogenic origin.

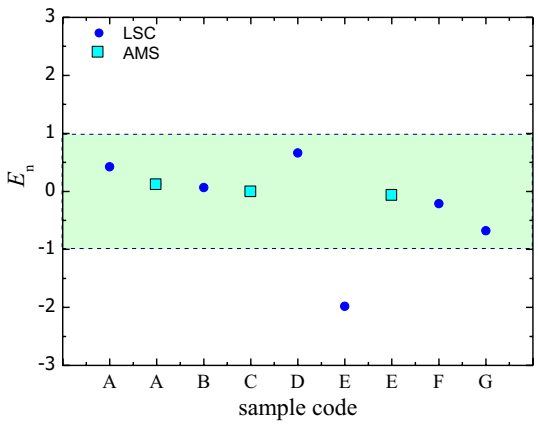

Fig. 2 The E_n values (Eq. 5) in ILC samples determined at the RBI by the two measurement methods, LSC (direct method by liquid scintillation counting) and AMS (accelerator mass spectrometry). The LSC result for sample C was not reported due to low SQP(E) value. The LSC result for sample E is the only non-acceptable result, due to the SQP(E) value close to the limit of the method applicability

Such a sample was an ideal one for validation of the direct LSC method for two reasons. First question was whether mixing of the highly-quenched liquid sample with a ^{14}C-free non-quenched sample can help in avoiding or diminishing problem of quenching. The second question was related to determination the SQP(E) value separating regions of qualitative and quantitative applicability of the direct LSC method. In addition, we compared the results related to volumetric and gravimetric determination of the UEO fraction.

The UEO was mixed with the ^{14}C-free petroleum ether sample (laboratory code Z-6266), which has been used as the background sample ($f_{bio} = 0$ %) with good quenching properties (SQP(E) = 864). The total mixture volume was 10 ml and 10 ml of Ultima Gold F scintillation cocktail was added, i.e., the measurement was performed in the same manner as all other organic liquid samples [13]. Changes in the SQP(E), count rate and f_{bio} values in UEO-petrol mixtures in the concentration range 0–100 % were monitored.

Volumetric determination of the UEO fraction in the mixture showed higher fluctuations in results than the gravimetric one due to lower uncertainty of weighing and occasional mistakes in pipetting sample volume portions. Further results are shown here based on the mass m of the UEO fraction.

The relations of the SQP(E) and count rates values are presented in Fig. 3a and b as functions of the mass of the UEO in the mixtures, while the f_{bio} is shown as a function of SQP(E) values in Fig. 3c. Mixtures containing $m < 2$ g of UEO (10 % and 20 % of UEO) gave SQP(E) values of 773 and 759 (Fig. 3a). The count rate of the mixtures increased first due to higher fraction of modern UEO (Fig. 3b) and after reaching a maximum started decreasing, although the fraction of modern sample was higher, but at the same time the higher fraction of dark modern sample caused higher quenching and lower SQP(E) values. The f_{bio} was determined to be 104.0 ± 1.2 % and 101.1 ± 1.3 %, respectively, for 10 % and 20 % mixtures, in accordance with the AMS result, shown as a line in Fig. 3c. Mixtures containing $2 \text{ g} < m < 5$ g (30–50 % of UEO) resulted in SQP(E) values between 671 and 609, while the f_{bio} values were approximately 150 %, i.e., qualitatively acceptable results. Mixtures containing more than 5 g of UEO (> 60 % of UEO) had SQP(E) values below 600. All these data taken together indicated that for SQP(E) values above approximately 700 the direct-LSC method at the RBI can give quantitatively good results for f_{bio}, as shown by the shaded areas in Fig. 3, while in the SQP(E) region between 700 and 600 the f_{bio} values can be taken as qualitative results.

It was also demonstrated that dilution of highly-quenched biogenic sample with non-quenched ^{14}C-free petroleum ether can give acceptable results in a certain region of mixing ratios. In future, more tests are planned, where different ^{14}C-free liquids will be used and also various

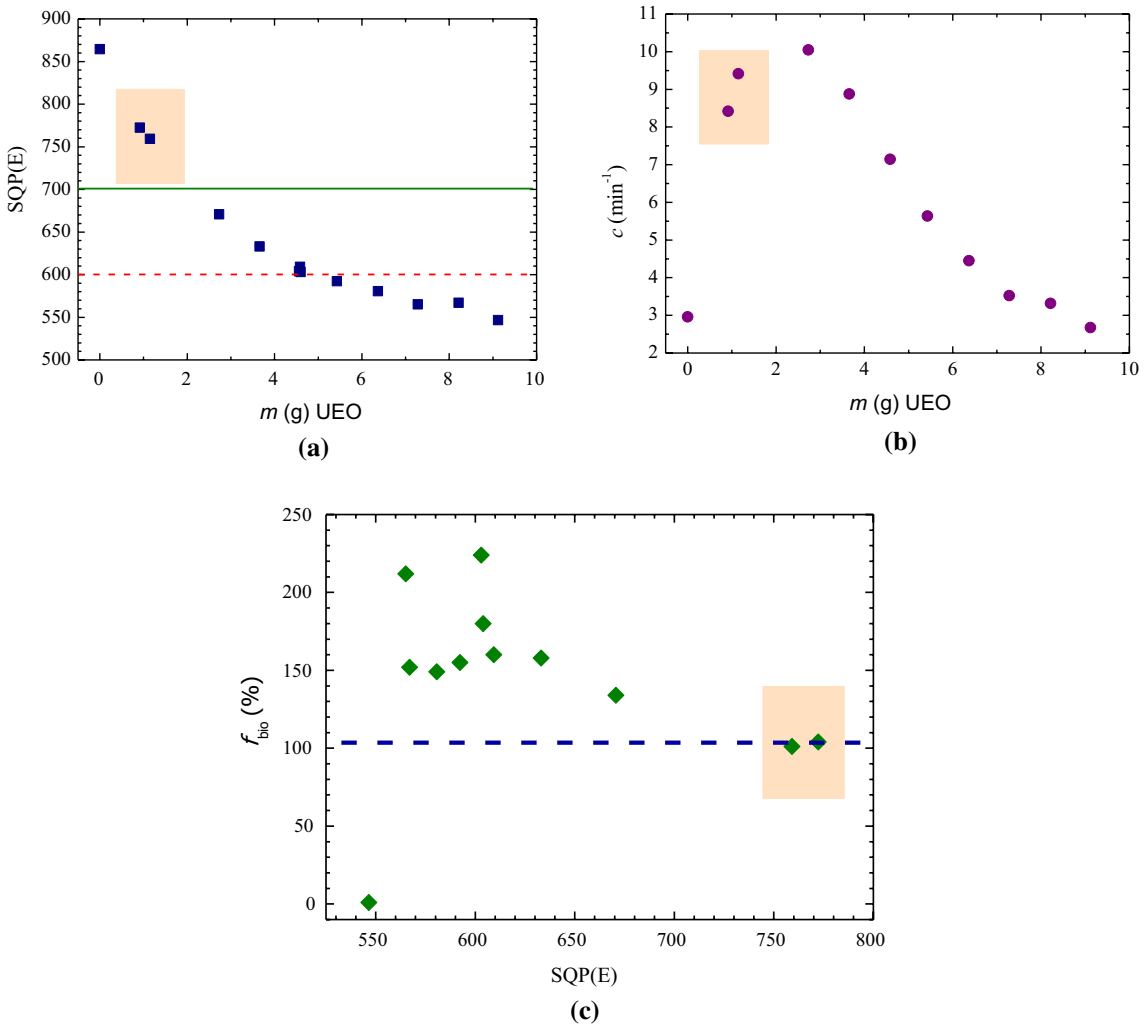

Fig. 3 **a** SQP(E) values in mixtures of UEO as a function of the mass of UEO. Lines at SQP(E) values of 600 and 700 represent the limits of qualitative and quantitative applicability, respectively. **b** Dependence of count rate on the mass of UEO. **c** Dependence of f_{bio} on SQP(E) values of the used edible oil in the mixture. The line represents the AMS results. Shaded areas in all panels represent samples in the quantitative region of f_{bio} determination

highly-quenched liquids, not necessarily modern ones. Also, the steps in mixture concentrations should be smaller than 10 %. The reference f_{bio} value can be the value determined by the AMS technique.

Conclusions

The presented results showed that the direct LSC method with an evaluation method developed in our laboratory was suitable for determination of the f_{bio} in liquid fuels, providing the correctly defined limits of applicability for highly quenched samples. We defined the limits of applicability of the direct-LSC method for both quantitative [SQP(E) > 700] and qualitative results [600 < SQP(E) < 700]. The RBI Laboratory has a possibility of applying also AMS ^{14}C measurement technique that can satisfactorily determine f_{bio} also in highly quenched samples as well, but the AMS technique is more time-consuming and more expensive than the direct LSC method.

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