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# BIO-BASED CARBON CONTENT DETERMINATION IN SOLID MATERIALS BY LIQUID SCINTILLATION COUNTING TECHNIQUE AT GLIWICE 14C AND MASS SPECTROMETRY LABORATORY

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In this research, tire rubber and its pyrolysis products, disposable packaging materials, and technical carbon black samples were analyzed for their $^{14}C$ isotope content and bio-based carbon using the liquid scintillation counting (LSC) technique. Pyrolytic oil and recovered carbon black showed higher $^{14}C$ concentrations with greater truck tire input, while passenger car rubber contained more $^{14}C$ than truck rubber. All packaging samples exceeded 100 pMC, confirming modern biomass origin, with wooden cutlery showing especially high values. In contrast, technical carbon black contained <1 pMC, indicating fossil-based production with little or no renewable input.

*Keywords:* radiocarbon methods, bio-based carbon content, liquid scintillation counting, environmental sustainability

## 1. INTRODUCTION

The radioactive isotope of carbon $^{14}C$, (radiocarbon) is the heaviest and an unstable naturally occurring isotope of carbon. It is relatively rare, accounting for only about ~$1\times10^{-10}$ % of all carbon, in contrast to the much more abundant stable isotopes: approximately 98.89% $^{12}C$ and 1.11% $^{13}C$ [1].

Radiocarbon ($^{14}C$) is produced naturally through interactions between cosmic rays and atmospheric nitrogen atoms, after which it immediately enters the global carbon cycle along with the stable isotopes of carbon ($^{12}C$ and $^{13}C$) [2]. A $CO_2$ molecule containing $^{14}C$ exhibits the same chemical properties as $CO_2$ containing only stable isotopes. Consequently, $^{14}CO_2$ is assimilated by plants through photosynthesis and subsequently transferred to animals via plant consumption, just like ordinary $CO_2$.

In the oceans, $^{14}C$ becomes incorporated into inorganic carbon species ($CO_2$, $HCO_3^-$, and $CO_3^-$) through molecular exchange across the air–sea interface, thereby entering the marine biosphere as well. Because all living organisms incorporate $^{14}C$ atoms, this isotope serves as a powerful tracer, providing a wide range of applications for the radiocarbon method [3, 4].

Radiocarbon ($^{14}C$) is widely applied as a dating tool for determining the age of carbon-containing materials and as an environmental tracer for studying carbon dynamics in natural systems [5]. The pioneering work on the measurement and application of $^{14}C$ in radiometric dating is credited to W. F. Libby and co-workers [6, 7]. The isotope has a half-life of 5700 ± 30 years [8]; thus, after approximately ten half-lives, radiocarbon is undetectable in a sample [9].

Beyond dating applications, the radiocarbon method has proven to be a reliable approach for determining the bio-based content of various products. In this method, $^{14}C$ concentration is expressed as percent modern carbon (pMC), while the bio-based carbon fraction is determined according to international standards, which apply an atmospheric correction factor (REF) corresponding to the reference year for 100% bio-based carbon.

The growing demand for energy and the generation of waste are two major challenges in the developing world. Among different waste streams, plastics and rubber pose the greatest treatment difficulties, together accounting for 31.2% of total waste [10].

In 2020, Europe generated 24.5 million tonnes of plastic waste. Only 14% was recycled, yielding 3.5 million tonnes of recyclates, while half was incinerated for energy recovery releasing about 95 million tonnes of $CO_2$ annually. To address this, the European Commission has set a target to eliminate plastic pollution by 2040 through a circular economy focused on reuse, recycling, and responsible disposal. It also aims to achieve full carbon neutrality and circularity by 2050 [11].

Rubber waste is equally concerning. Over 1.7 billion tires are produced globally each year [12], including about 4.2 million tonnes in the EU in 2020 [13]. According to [10], two-thirds of used tires end up in landfills or are illegally dumped, creating severe risks to both the environment and human health. Waste tires now represent one of the most pressing global environmental issues, as rubber makes up a significant share of solid waste.

This study collects and analyses the results obtained by the LSC technique published in several previous publications [14-16]. The focus was placed on tire rubber and its pyrolysis products, technical carbon black (TCB), and disposable packaging materials (DPMs) all of which are significant in recycling processes and sustainable production strategies. The primary aim was to assess the modern carbon content of these materials by liquid scintillation counting (LSC), in order to evaluate compliance with environmental protection regulations designed to promote green technologies and mitigate greenhouse gas emissions.

## 2. MATERIALS

In this study, several categories of every day-use product were analyzed. The first group included tire rubber and pyrolysis products, such as recovered carbon black and pyrolytic oil, provided by Contec Inc., a Warsaw based company employing tire pyrolysis to produce eco-friendly alternatives to virgin carbon black and petrochemical feedstock, while addressing the global end-of-life tires waste. Samples were supplied in three batches, each containing a mixture of shredded truck and passenger car tires in varying proportions, with the corresponding pyrolysis oil and recovered carbon black obtained from the same batch.

The second group comprised ten types of disposable packaging materials (DPMs) collected from Quick Pack, Vigo, and Bio-pack. These included paper plates, boxes, paper straws, cups, baking paper, sugarcane-based plates and boxes, wheat bran plates, and wooden cutlery.

Table 1. Description of the samples analyzed.

Table 1. Opis analizowanych próbek.

| No. | Sample Name | Sample code | Description |
|---|---|---|---|
| 1 | Pyrolytic oil-1 | CONT-1 | Unknown mixture of truck and passenger car tires. |
| 2 | Pyrolytic oil-2 | PCO-2 | 100% passenger car tires. |
| 3 | Recovered carbon black-1 | rCB-2 | 100% passenger car tires. |
| 4 | Rubber-1 | CR-2 | 100% passenger car tires. |
| 5 | Pyrolytic oil-3 | TCO-3 | 70% truck and 30% passenger car tires. |
| 6 | Recovered carbon black-2 | rCB-3 | 70% truck and 30% passenger car tires. |
| 7 | Rubber-2 | CR-3 | 70% truck and 30% passenger car tires. |
| 8 | Disposable plates-1 | QSCP-1 | In white, a kind of paper disposable plates, made from sugarcane fibers. |
| 9 | Disposable cups | QPC-2 | Pale white, made with paper stuff. |
| 10 | Baking paper | QBP-3 | In brown color, also known as vegetable parchment; cellulose-based composite. |
| 11 | Disposable straws | QPS-4 | Brown on the outside and white on the inside, made of paper material. |

| | | | |
|---|---|---|---|
| 12 | Disposable plates-2 | QPP-5 | In white color, made of paper material. |
| 13 | Wooden cutlery | QWC-6 | Consists of wooden spoons, wooden forks, and wooden knives. |
| 14 | Disposable plates-3 | QBPP-7 | In brown color, made of paper. |
| 15 | Disposable plates-4 | VWBP-8 | Brown in color made from wheat bran. |
| 16 | Disposable boxes | BSCB-9 | Pale white, made from sugarcane bagasse. |
| 17 | Disposable plates-5 | BPP-10 | Pale white, made from paper. |
| 18 | Technical carbon black-1 | TCB-N330 | Surface area of 76-80 $m^2/g$ with medium-high reinforcement. Compounds used for tire tread, carcass, and sidewalls. |
| 19 | Technical carbon black-2 | TCB-N550 | Surface area 39-41 $m^2/g$ with medium-high reinforcement. It is used in the inner liners, carcasses, and sidewalls of passenger tires and rubber goods. |
| 20 | Technical carbon black-3 | TCB-N660 | Surface area 34-36 $m^2/g$, medium reinforcement. It is used in inner liners, sidewalls, sealing rings, cable jackets, rubber molding, and extruded goods. |
| 21 | Technical carbon black-4 | TCB-N772 | Surface area 31-32 $m^2/g$, with semi-reinforcement. They are used as inner liners in tires and rubber items. |

The third group consisted of technical carbon black (TCB) samples, also provided by Contec Inc. Four graded TCB samples were examined, classified according to surface area, aggregate morphology at the nanoscale, ash content, and other chemical characteristics [17, 18]. Table 1 describes all the sample categories in detail.

## 3. METHODOLOGY

### 3.1. Liquid Scintillation Counting (LSC) measurements

Bio-based carbon in tire rubber, tire pyrolysis products, disposable packaging, and TCB samples was determined by LSC technique at the Gliwice Radiocarbon Laboratory (Institute of Physics, Division of Geochronology and Environmental Isotopes). Measurements followed benzene synthesis using analytically pure reagents. Glassware was cleaned with deionized water and dried at 80 °C. Benzene was synthesized directly from samples (except tire rubber, washed with hot deionized water) [14].

The Gliwice laboratory operates a dedicated benzene line with fume extractors and hoods for safe handling. Benzene synthesis included:

#### 3.1.1 $CO_2$ and Carbide Synthesis

Solid samples were reacted with metallic lithium (1:1) in a purified, hermetically sealed reactor at 700 °C under pressure control. The reactor was pre-cleaned and heated at 700 °C in a muffle oven.

#### 3.1.2 Pyrolytic Oil Processing

Pyrolytic oil was analyzed in two steps. First, the oil was evaporated in a small inner reactor onto heated lithium in an outer reactor. After cooling, residues from the inner reactor were transferred to the outer reactor and treated like solid samples with added lithium. Reaction times varied by material: ~ 1 h for TCB, ~ 40 min for pyrolytic oil, and 1–2 h for tire rubber[14].

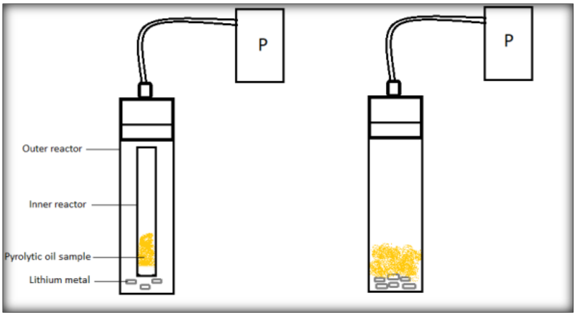


Fig. 1. Processing of Pyrolytic oil in metal reactor.

Rys. 1. Przetwarzanie oleju pirolitycznego w reaktorze metalowym

### 3.1.3 Carbonization of Disposable Packaging Material

For disposable packaging materials, samples were first carbonized in a cleaned metal reactor with a small hole to limit air access. Carbonization was carried out in a muffle furnace at 700 °C under controlled pressure for 7–10 minutes. After cooling, the carbonized material was ground into powder and directly treated with lithium metal [15].

### 3.1.4 General Reaction of Carbide Synthesis

$$2CO_2 + 10Li \xrightarrow{700\ ^\circ\mathrm{C}} Li_2C_2 + 4Li_2O \qquad \text{Eq. 1}$$

### 3.1.5 Synthesis and Purification of Acetylene

A separate line is used for the production of acetylene by the hydrolysis of $Li_2C_2$.

$$Li_2C_2 + H_2O \xrightarrow{\text{Room temperature}} C_2H_2 + 2LiOH \qquad \text{Eq. 2}$$

Acetylene was purified using a hygroscopic trap ($K_2Cr_2O_7 + H_2SO_4$), and the pure gas was collected in a liquid nitrogen cooled glass trap.

### 3.1.6 Benzene Synthesis and Purification

$$3C_2H_2 \xrightarrow{\text{Room temperature}} C_6H_6 \qquad \text{Eq. 3}$$

Benzene was synthesized from acetylene via trimerization on a chromium catalyst (preheated at 600 °C for 30 min). It was purified from water vapor and radon: dried over sodium metal overnight and stored at -15 °C for one month to allow radon decay ($T_{½}$ = 3.8 days).

After purification, 2 ml of benzene was mixed with butyl-phenyl-bipheny-oxydiazole (butyl-PBD) scintillator for activity measurement. $^{14}C$ activity was determined using a Quantulus 1220™ liquid scintillation $\beta^-$ spectrometer. Polish Analar-grade benzene was used as background, and ANU-sucrose served as the standard [19].

Fig. 2 Benzene line at Gliwice $^{14}C$ and Mass Spectrometry Laboratory.

Rys. 2. Linia do przetwarzania próbek na benzen w Laboratorium $^{14}C$ i Spektrometrii Mas w Gliwicach

## 3.2. Isotope Ratio Mass Spectrometry (IRMS)

Isotope ratios were determined using an **IsoPrime 100** spectrometer (continuous flow) coupled with a **MultiFlow system** and **Gilson GX-271 autosampler**. The MultiFlow system enabled automatic collection and transfer of $CO_2$ from sample vessels to the spectrometer, with gas separated by chromatography before isotope analysis [20, 21].

IRMS was used to calculate isotope fractionation corrections for LSC measurements [22]. Benzene produced for LSC was also used for IRMS: ~ 5 μl of benzene was sealed in Simax tubes with ~300 mg CuO, combusted at 560 °C for 12 h, and $CO_2$ was collected in a cold trap (liquid $N_2$) after passing through a water vapor trap. After pressure controlled quantification, three $CO_2$ subsamples were transferred into MultiFlow vessels for analysis.

IAEA reference materials (NBS-22 oil, IAEA-CH-6 sucrose and IAEA-C3 cellulose) were used as standards and were subjected to the same preparation as the tested samples.

Radiocarbon concentrations were corrected using the measured $\delta^{13}C$ values according to [22]

$$A_{SN} = A_S \left[1 - \frac{2(25+\delta^{13}C)}{1000}\right]. \qquad \text{Eq. 4}$$

## 4. RESULTS AND DISCUSSION

$^{14}C$ concentrations are expressed as percent modern carbon (pMC) [22] and bio-based carbon content according to EN16640 [23], as listed in Table 2. A reference value of 100% bio-based carbon (pMC) for the year 2022 was used, based on University of Groningen data [24, 25]. For tires and pyrolysis products, a reference year of 2011 (103.3 pMC) was applied, reflecting the typical 10–15 year disposal age. $\delta^{13}C$ values (‰ VPDB) were used for standardized isotope fractionation correction in LSC measurements.

Pyrolytic oil from truck tires showed $^{14}C$ concentrations of 50.35 ± 0.49 pMC, while passenger car tires gave 43.90 ± 0.44 and 41.30 ± 0.47 pMC. An unknown tire mixture yielded 41.95 ± 0.37 and 43.43 ± 0.38 pMC. Recovered carbon black (rCB) had much lower values: 5.08 ± 0.18 pMC (car tires) and 6.89 ± 0.18 pMC (truck tires)[14].

Table 2. Results of $^{14}C$ concentrations, $\delta^{13}C$ measurements, and calculated bio-based carbon content.

Tabela 2. Wyniki oznaczeń stężenia $^{14}C$, pomiarów $\delta^{13}C$ oraz obliczonej zawartości węgla pochodzenia biologicznego.

| **No.** | ***Sample*** | ***Lab code*** | ***$^{14}C$ concentration (pMC)*** | ***$\delta^{13}C_{VPDB}$ (‰)*** | ***Bio-based carbon content (%)*** |
|---|---|---|---|---|---|
| 1 | *CONT-1 Pyrolytic Oil* | *GdS-4257* | *41.95 ± 0.37* | *-27.53 ± 0.07* | *40.6 ± 0.36* |
| | | *GdS-4258* | *43.43 ± 0.38* | | *42.0 ± 0.37* |
| 2 | *PCO-2 Pyrolytic Oil* | *GdS-4320* | *41.30 ± 0.47* | *-27.2 ± 0.07* | *40.0 ± 0.45* |
| | | *GdS-4365* | *43.90 ± 0.44* | | *42.5 ± 0.43* |
| 3 | *TCO-3 Pyrolytic Oil* | *GdS-4373* | *50.35 ± 0.49* | *-26.57 ± 0.10* | *48.7 ± 0.47* |
| 4 | *rCB-2 Recovered Carbon Black* | *GdS-4327* | *5.08 ± 0.18* | *-26.83 ± 0.18* | *4.9 ± 0.17* |
| 5 | *rCB-3 Recovered Carbon Black* | *GdS-4394* | *6.89 ± 0.18* | *-26.48 ± 0.04* | *6.7 ± 0.17* |
| 6 | *CR-2 Rubber* | *GdS-4337* | *15.54 ± 0.23* | *-26.69 ± 0.07* | *14.8 ± 0.22* |
| 7 | *CR-3 Rubber* | *GdS-4339* | *13.59 ± 0.23* | *-26.14 ± 0.18* | *12.9 ± 0.22* |
| 8 | *TCB-N330* | *GdS-4543* | *0.589 ± 0.084* | *-32.44 ± 0.11* | *0.589 ± 0.084* |

| | | | | | |
|---|---|---|---|---|---|
| 9 | *TCB-N550* | *GdS-4552* | *0.189 ± 0.063* | *-30.41 ± 0.072* | *0.189 ± 0.063* |
| 10 | *TCB-N660* | *GdS-4554* | *0.908 ± 0.092* | *-31.82 ± 0.44* | *0.908 ± 0.092* |
| 11 | *TCB-N772* | *GdS-4555* | *0.124 ± 0.091* | *-31.45 ± 0.020* | *0.124 ± 0.091* |
| 12 | *QSCP-1 Sugarcane plates* | *GdS-4610* | *103.20 ± 0.74* | *-24.16 ± 0.059* | *103.20 ± 0.74* |
| 13 | *QPC-2 Paper cups* | *GdS-4611* | *114.65 ± 0.80* | *-29.4 ± 0.037* | *114.65 ± 0.80* |
| 14 | *QBP-3 Baking paper* | *GdS-4616* | *109.99 ± 0.78* | *-27.43 ± 0.23* | *109.99 ± 0.78* |
| 15 | *QPS-4 Paper straws* | *GdS-4617* | *110.36 ± 0.78* | *-26.97 ± 0.36* | *110.36 ± 0.78* |
| 16 | *QPP-5 Paper plates* | *GdS-4620* | *108.30 ± 0.72* | *-26.14 ± 0.21* | *108.30 ± 0.72* |
| 17 | *QWC-6 Wooden cutlery* | *GdS-4618* | *125.51 ± 0.87* | *-30.02 ± 0.16* | *125.51 ± 0.87* |
| *18* | *QBPP-7 Brown paper plates* | *GdS-4621* | *111.36 ± 0.74* | *-27.78 ± 0.24* | *111.36 ± 0.74* |
| *19* | *VWBP-8 Wheat plates* | *GdS-4622* | *103.56 ± 0.71* | *-14.24 ± 0.74* | *103.56 ± 0.71* |
| *20* | *BSCB-9 Sugar cane boxes* | *GdS-4623* | *102.45 ± 0.70* | *-15.84 ± 0.42* | *102.45 ± 0.70* |
| *21* | *BPP-10 Paper plates* | *GdS-4624* | *114.92 ± 0.76* | *-29.68 ± 0.16* | *114.92 ± 0.76* |

$^{14}C$ concentrations in pyrolytic oil increased with the proportion of truck tires in the feed. In contrast, rubber samples did not follow this trend: passenger car tire rubber showed higher $^{14}C$ content than truck tire rubber. This likely reflects the complex composition of tires, which combine natural (bio-based) and synthetic (fossil-derived) rubbers in different layers. Thus, a small rubber sample represents only its specific tire location rather than the whole tire or batch subjected to pyrolysis[14].

All of the four different graded technical carbon black samples showed very low $^{14}C$ concentrations (<1 pMC), indicating a predominantly fossil origin and production prior to the introduction of biogenic materials[16].

All disposable packaging samples showed $^{14}C$ concentrations above 100 pMC, confirming their modern origin and supporting their classification as green recyclables. Nine of the ten samples ranged between 102.45 ± 0.70 and 114.92 ± 0.76 pMC. Paper-based materials (six samples) gave values of 108.30 ± 0.72 to 114.92 ± 0.76 pMC, with $\delta^{13}C$ values from -29.68 ± 0.16‰ to -26.14‰, consistent with literature data [15, 26-28]. The wheat bran plate (VWBP-8) showed 103.56 ± 0.71 pMC and a $\delta^{13}C$ value of -14.24 ± 0.74‰, characteristic of C-4 plants [15]. Sugarcane samples, the plate (QSCP-1) and box (BSCB-9), yielded 102.45 ± 0.70 pMC and 103.20 ± 0.74 pMC, with $\delta^{13}C$ values of -24.16 ± 0.06‰ and -15.84 ± 0.42‰, respectively. The $\delta^{13}C$ value of BSCB-9 corresponds to wheat bran, while the unexpected QSCP-1 result may be due to a plastic coating layer. The wooden cutlery sample (QWC-6) showed the highest concentration at 125.51 ± 0.87 pMC, with a $\delta^{13}C$ value of -30.02 ± 0.16‰, consistent with reported values for wood [15, 29].

## CONCLUSION

The LSC technique was tested at the Gliwice $^{14}C$ and Mass Spectrometry Laboratory for determining $^{14}C$ concentrations in tire rubber and its pyrolysis products, technical carbon black (TCB), and disposable packaging materials from different manufacturers. The method proved suitable and reliable for all types of materials studied. Bio-based carbon content was calculated according to EN16640 (2017) using reference values (REF). In tire samples, $^{14}C$ concentrations were higher in pyrolytic oil than in rubber, and also higher in truck tires than in passenger car tires. Contrary to expectations, all TCB samples contained only trace amounts of $^{14}C$ (<1 pMC), indicating they were produced almost entirely from fossil sources and likely manufactured before the incorporation of biogenic material into the production processes. All disposable packaging samples showed $^{14}C$ concentrations above 100 pMC, confirming their modern biomass origin. The wooden sample showed the highest values, reflecting the contribution of older trees. This work also supports green recycling by using renewable resources in the production of various materials and helping to reduce greenhouse gas emissions.

## SYMBOLS – OZNACZENIA

$A_S$ – value in equation (4) the measured $^{14}C$ concentration

$A_{SN}$ – value in equation (4) the corrected $^{14}C$ concentration

$T_{½}$ – half life

$\delta^{13}C$ – delta 13 carbon isotope

‰ – permille

## ABBREVIATIONS – SKRÓTY

CB – Carbon Black

DPM – Disposable Packaging Material

ELT – End-of-Life Tires

GdS – Gliwice Laboratory code for LSC technique

IAEA – International Atomic Energy Agency

IRMS – Isotope Ratio Mass Spectrometry

LSC – Liquid Scintillation Counting

rCB – Recovered Carbon Black

TCB – Technical Carbon Black

VPDB – Vienna Peedee Belemnite

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# OZNACZANIE ZAWARTOŚCI WĘGLA POCHODZENIA BIOLOGICZNEGO W MATERIAŁACH STAŁYCH TECHNIKĄ CIEKŁOSCYNTYLACYJNĄ W LABORATORIUM $^{14}C$ I SPEKTROMETRII MAS W GLIWICACH

W niniejszych badaniach analizowano zawartość izotopu $^{14}C$ oraz udział węgla pochodzenia biologicznego w gumie oponowej i jej produktach pirolizy, jednorazowych materiałach opakowaniowych oraz w próbkach technicznej sadzy, z wykorzystaniem techniki ciekłoscyntylacyjnej (LSC). Olej pirolityczny i odzyskana sadza wykazywały wyższe stężenia $^{14}C$ przy większym udziale opon ciężarowych, podczas gdy guma z opon osobowych zawierała więcej $^{14}C$ niż guma z opon ciężarowych. Wszystkie próbki opakowaniowe przekraczały wartość 100 pMC, co potwierdza ich pochodzenie z nowoczesnej biomasy, przy czym szczególnie wysokie wartości odnotowano w przypadku sztućców drewnianych. Dla kontrastu, techniczna sadza zawierała <1 pMC, co wskazuje na produkcję opartą na surowcach kopalnych, z niewielkim lub zerowym udziałem odnawialnych źródeł.

*Słowa kluczowe:* metoda radiowęglowa, zawartość węgla pochodzenia biologicznego, ciekłoscyntylacyjne zliczanie promieniowania, zrównoważony rozwój środowiskowy

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