

GRAVIMETRIC AND INSTRUMENTAL METHODS COMPARISON FOR EXPERIMENTAL DETERMINATION OF CARBONATE CARBON CONTENT IN SOLID MINERAL FUELS

by

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The content of combustible elements in solid mineral fuels (carbon, hydrogen, etc.) are very important, since they most directly affect the heat value. It should be noted that the fuel heat value depends on many other constituents, such as ash and moisture. In this paper, special attention has been paid to carbon content. In solid mineral fuels, carbon is found alone or bound in the form of various compounds. One of them is mineral carbonate compounds bound as carbonate carbon, which originates from absorbed CO₂ from atmosphere. Determination of carbonate carbon content of solid mineral fuels was performed by standard gravimetric method (according to ISO 925: 2019), and newly developed instrumental method, using thermogravimetric analyzer LECO TGA 701. Comparison of obtained experimental results was done. Four types of coal, Kolubara lignite, Kostolac lignite, brown coal, and control coal sample were included in experimental analysis. In addition, moisture in the samples was also determined using analytical method and inspected using LECO TGA 701 thermogravimetric analyzer, as well as total carbon content using the LECO CHN 628 elemental analyzer. An analysis and comparison of the obtained results was performed, and comments and conclusions are presented. The experiments were done in the department for fuel characterization, Laboratory for Thermal Engineering and Energy, Institute of Nuclear Sciences Vinča.

Key words: carbonate carbon, solid mineral fuels, proximate analysis, ultimate analysis, total carbon content

Introduction

In nature carbon, C, occurs free, in elemental form, or bound in the form of compounds. Elemental carbon occurs in the form of its allotropic modifications, among which graphite and diamond are the most known. Bound carbon is found within organic or inorganic compounds. In an inorganic form, it can be found as an integral part of rocks, to form carbonate mineral compounds, such as CaCO₃, MgCO₃, etc. This is the so-called carbonate carbon, which comes from absorbed CO₂ from the atmosphere.

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The carbon is best known as a constituent of fossil fuels, coal, oil and natural gas, which are formed from plant and animal residues deposits under high pressure. In fossil fuels, carbon is the most important chemical element, since heat value mostly depends on carbon and hydrogen content. These elements are components of every fossil fuel.

Since the paper deals with solid fossil fuel-coal, the basic characteristics, types and composition of coal will be listed. Coal is classified according to several criteria: origin, age, purpose, heat value, *etc.*

Combustion of carbon from fossil fuels, particularly coal, releases the product of combustion – CO₂. The CO₂ is a gas under standard conditions. It is a colorless and odorless, heavier than air, does not burn and does not support combustion. The three main sources of CO₂ are: natural sources-volcanoes, burning fossil fuels and passenger, and transport vehicles with internal combustion engines. In previous period, a balance has been established between the amount of CO₂ emitted by volcanoes and the amount of CO₂ consumed during photosynthesis, but in recent times, the combustion of fossil fuels in thermal power and industrial plants, has disturbed the balance, and consequently there is more CO₂ in atmosphere than nature can absorb. The increased concentration of CO₂, as a direct GHG, and its ability to absorb infrared radiation, leads to additional atmosphere and Earth's surface warming, thus creating a greenhouse effect [1]. This ultimately leads to climate change on Earth.

Lignite is characterized by a preserved woody structure with dark tonality. Lignite from The Republic of Serbia have carbon content in range of 30-35% by mass or even more, while heat value range from 6-12.5 MJ/kg. Brown coal have brown to black color, with carbon content of 35% and more, while its heat value is in the range of 16.7-29.3 MJ/kg. Brown coal is followed by hard coal, with anthracite being its last form. For example. hard coal has a moisture content of 3-10% and carbon content in the range of 86-97% [2], while anthracite is characterized with the highest heat value of 35.6-37.7 MJ/kg.

Lignite is a major energy resource in Central and Southeast Europe. Republic of Serbia have low quality lignites, with a high moisture content and low calorific value. Main lignite deposits in Republic of Serbia are open pit mine Kolubara, located about 60 km from Belgrade, with an area of about 600 km², and Kostolac open pit mine, located about 90 km from Belgrade and covers an area of 145 km² [3]. The Kolubara Basin participates with 65% in total coal production [4].

Experimental laboratory tests of lignite samples from open-pit mines Kolubara and Kostolac which included ultimate analysis, proximate analysis, calorific value, *etc.*, in order to determine carbon emission characteristics are presented [5, 6].

Brown coal has a high heat value and low moisture content, having widespread use. By heating without the presence of air, it releases volatile substances that form a new hard, black and porous product-coke.

The aim of this paper is testing of newly established instrumental method using TGA device, in order to determine carbonate carbon content, with the results obtained by standard gravimetric method. The comparison of the results of both methods was performed in the department for fuel characterization, Laboratory for Thermal Engineering and Energy, Institute of Nuclear Sciences Vinča.

Materials and methods

Sample preparation

In experimental laboratory tests for determining the content of carbonate carbon in solid fuels-coals, a total of six samples of different types were tested. Two samples of Kolubara

lignite, two samples of Kostolac lignite, one sample of brown coal and one control sample-coal from the proficiency test scheme were analyzed. Tested samples summary is presented in tab. 1.

Table 1. Tested coal samples

Sample label	Description
Kolubara 1	Kolubara lignite, February, 2020
Kolubara 2	Kolubara lignite, March, 2020
Kostolac 1	Kostolac lignite, May, 2020
Kostolac 2	Kostolac lignite, July, 2020
Brown coal	Brown coal, May, 2020
Control coal	Control coal sample, 2015

Sample preparation, moisture content determination in analytical sample, was performed according to standard [7]. Each coal sample is prepared so that the moisture content of the sample is in balance with the atmosphere moisture. Sample preparation process included crushing, grinding, mixing and weight reduction by dividing the sample operations, after which an analytical sample was ready for further analyzes.

Gravimetric determination of the analytical moisture

Gravimetric analysis includes a set of methods of analytical chemistry for the quantitative determination of ingredients based on mass. The determination of the analytical moisture was determined by measuring the mass of the sample before and after drying in an oven at 105 °C. Weight an empty vessel and record its mass, then weigh about 1 g of coal sample and record the value. Measured sample is dried in an oven for a period of two hours. After two hours, dish was removed from oven, left in desiccator to cool, re-measured and the mass of the dish was recorded.

Analytical moisture is determined:

$$W_a = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where m_1 [g] is the mass of empty container with lid, m_2 [g] – the mass of the container with lid and coal sample before drying, and m_3 [g] – the mass of the container with lid and coal sample after drying, in grams.

Gravimetric determination of carbonate carbon content

Determination of carbonate carbon content for these coal samples was performed using the analytical gravimetric method according to ISO 925: 2019 [8]. According to analytical gravimetric method, chemicals with known purity degree, tab. 2, and distilled water were used. The assembled apparatus [8] consists of a reaction balloon connected to a condenser, a separating funnel, absorption columns, a pump as well as a flow meter, as shown in fig. 1.

Table 2. Analytical gravimetric method, used chemicals

Chemical compound	Name	Molar mass [g·mol ⁻¹]	CAS number	Source
Mg(ClO ₄) ₂	Magnesium perchlorate	223.206	10034-81-8	Fisher
NaOH	Sodium hydroxide	39.997	1310-73-2	Mallinckrodt Baker
CuSO ₄ ·5H ₂ O	Copper sulfate pentahydrate	249.68	7758-99-8	Posh S. A.
HCl	Hydrochloric acid	36.458	7647-01-0	Macron fine chemicals

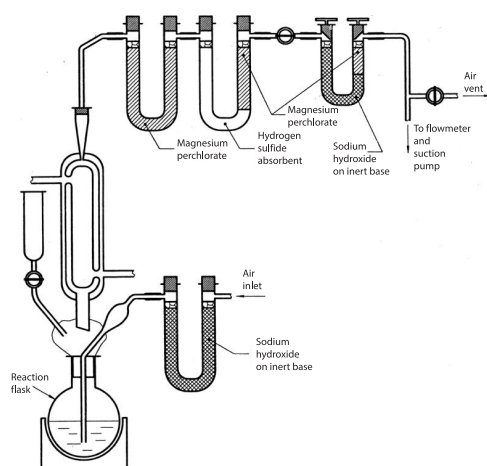


Figure 1. Experimental apparatus according to ISO 925:2019 [8]

At the bottom, the apparatus is connected to a purification column, opened on one side, and filled with calcium carbonate in order to bind CO_2 . There are three absorption columns on the upper side of the apparatus. The first column is filled with $\text{Mg}(\text{ClO}_4)_2$ to absorb moisture, the second one is filled with $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in order to remove hydrogen sulfide and the third column is filled with NaOH . The second and third absorption columns were also partially filled with a protective layer of $\text{Mg}(\text{ClO}_4)_2$.

The measurements were performed by transferring a certain mass of coal sample to the reaction flask. Ethanol and distilled water were added, closed and mixed well. In the next step reaction flask was assembled with the rest of the apparatus. Air was introduced through the apparatus at a certain flow rate for 10 minutes. The absorption column with NaOH was separated from the rest of the apparatus and weighed on the balance. After measurement, the absorption column is reconnected to the rest of the apparatus, and HCl is added to the reaction flask. The heating of the reaction vessel was achieved by means of a sand bath, during which the boiling temperature had to be reached in a period of 15 minutes and maintained for the next 30 minutes. The NaOH absorption column was re-measured after completion of the reaction and the resulting mass difference was used to calculate the carbonate carbon content and the carbon dioxide content for the tested coal sample.

The correctness of the apparatus should be checked after its assembly by using a reagent of known carbon or carbonate content. According to the standard ISO 925: 2019, two reagents can be used for verification, NaCO_3 and CaCO_3 . Both reagents were used to check the correctness of the apparatus and showed good agreement and repeatability according to the standard. However, when sodium carbonate was used on TGA 701, the obtained values of CO_2 content differed from CO_2 stoichiometric content, *i.e.* the values obtained using the gravimetric method. This is explained by the low melting point of sodium carbonate (851°C), close to the temperature regime in TGA 701 of 815°C . Having in mind this limitation, calcium carbonate was used to check the correctness of the apparatus, tab. 3. The apparatus is correctly assembled if the deviations between the stoichiometric and measured values do not differ by more than 10% (standard ISO 925: 2019).

After the correctness check, samples can be analyzed in order to determine carbon or CO_2 content. These two quantities can be expressed:

$$W_C = \frac{27.29m_2}{m_1} [\%] \quad (2)$$

The CO_2 content for the analyzed sample can be determined as a percentage by mass:

$$W_{\text{CO}_2} = \frac{100m_2}{m_1} [\%] \quad (3)$$

where m_1 [g] is the test sample mass, m_2 [g] – the increase in mass of absorption tube, W_C [%] – the carbonate carbon content, and W_{CO_2} [%] – the CO_2 content.

Table 3. Sodium and calcium carbonate, basic data

Chemical compound	Name	Molar mass [g mol^{-1}]	Melting point [°C]	CAS number	Source
NaCO ₃	Sodium carbonate	105.9888	851	497-19-8	Fisher
CaCO ₃	Calcium carbonate	100.0869	1339	471-34-1	Acros organics

Determination moisture and carbonate carbon content using the thermogravimetric analyzer

Thermogravimetric analyzer LECO TGA 701 is used for proximate analysis of organic, inorganic and synthetic materials by measuring the loss of mass as a function of time, temperature in a controlled environment of a given atmosphere. The device consists of a computer and a thermally insulated furnace with a refractory wall in which there is a rotating carousel capable to hold up to 19 crucibles (vessels) for samples that are going to be tested. After selecting the method for analysis, the vessels (crucibles) are placed in the furnace, filled with samples, during which the mass is automatically measured and stored in the computer's memory, since the device has a built-in balance. The carousel rotation and samples mass loss measurement in the furnace is continuous until the difference in mass between two consecutive measurements for each sample is less than the already programmed value. The introduction of technical gases, nitrogen, oxygen and air, necessary for the unit operation, is also automated.

For coal samples testing, temperature regime has been adjusted according to following guidelines: for moisture determination the device is programmed to measure the difference in the initial mass of samples and the mass of the samples after holding at 105 °C to a constant weight, heating rate was 5 °C per minute from 25-107° C with pure nitrogen atmosphere. For ash determination heating rate was 20 °C per minute from 107-510° C, atmosphere is a pure oxygen; and finally for CO₂ determination heating rate was 20 °C per min from 510-815 °C, atmosphere is a pure oxygen. All measurements were done in duplicate, and the result is the mean value of these measurements.

The correctness of set method for determination carbonate carbon content using TGA were checked using a reagent of known carbon or carbonate content. According to the standard ISO 925: 2019, CaCO₃ has been used for verification.

Determination of total carbon content

Ultimate analysis of total carbon content in coal samples were performed using the LECO CHN 628 analyzer by burning the samples, according to standard ASTM D5373-14:2014 [9]. The mass of the tested coal sample is measured on an analytical balance-the mass of the sample should be about 0.1000 g. The sample is placed on the opening on the carousel, after which the sample enters the device chamber located just above the furnace. The sample is then introduced into a primary furnace containing pure oxygen which leads to rapid and complete combustion of the sample. The combustion products are transferred to a secondary furnace for further oxidation and removal of particles. Using helium carrier gas, the combustion products are carried to an infrared cell used for the detection and analysis of water vapor and carbon dioxide, while nitrogen oxides are reduced as they passed through the reduction cell. In this way, total carbon, hydrogen and nitrogen content can be determined indirectly, based on the concentration of gases from the combustion products of the samples. Specifically, by burning

the sample, based on the total amount of the combustion products, for example carbon dioxide, or its concentration, and based on its known infrared radiation absorptivity, it is possible to determine the total carbon content in coal.

Results and discussion

According to ISO 925: 2019 [8], after assembling the apparatus presented on fig. 1, it is necessary to check the apparatus, *i.e.* the tightness, since there are many connections between glass tubes using silicone hoses, and *U*-tubes as well. Two compounds are used as test reagents for apparatus tightness: Na_2CO_3 or CaCO_3 .

The apparatus correctness at first has been checked with sodium carbonate. The deviation of measured value to theoretical (stoichiometric value) was not more than 4%. However, an issue with sodium carbonate occurred when determining the CO_2 content using an instrumental method with thermogravimetric analyzer LECO TGA 701. The analyzer was programmed to operate at a temperature of up to 815 °C. Since the sodium carbonate melting point is relatively low and is 851 °C, sodium carbonate began to decompose at much lower temperatures. Therefore, it was not possible to obtain comparable results of the gravimetric and instrumental methods. For this reason, calcium carbonate was used as a test reagent, since it has a higher melting point, 1339 °C, and therefore, did not decompose at temperatures below 800 °C.

Based on apparatus tightness verification for gravimetric method, a comparison of the stoichiometric value of CO_2 content and the values obtained by experiments by gravimetric and instrumental methods was performed, as can be seen in tab. 4. It is obvious that the set-up of the apparatus was performed correctly, which is indicated by fact that the deviation of measured fraction values of released CO_2 from calcium carbonate and stoichiometric values, are below 10%. Considering the successfully assembled apparatus, the determination of the carbonate carbon content of the listed samples in tab. 1 was ready to be performed and presented.

Table 4. Stoichiometric and experimental released CO_2 fraction from calcium carbonate obtained by gravimetric and instrumental methods

Sample mass [g]		Measured	Average	Stoichiometry	Deviation
		W_{CO_2} [%]	W_{avCO_2} [%]	W_{CO_2} [%]	[%]
Gravimetric method	Test 1	46.23	46.11	43.96	5.16
	Test 2	45.98			4.60
Instrumental method	Test 1	42.82	42.79		2.59
	Test 2	42.76			2.73

After successful correctness and tightness check of the assembled apparatus, moisture content was determined by analytical and instrumental method for proximity analysis, except for samples Kolubara 2 and Kostolac 2 where moisture was examined only by instrumental method. The results of moisture content by samples are shown in tab. 5. Determining the moisture of samples is important, since it is necessary to know on what basis other values and parameters are determined. Insight into the obtained experimental data, presented in tab. 5, shows the difference in certain values of moisture obtained using the two methods. This is not uncommon, since the methods are essentially different, so the observed value differences are not crucial. In the case of the analytical method, drying and determination of moisture are performed in the free air atmosphere, at atmospheric pressure, while for the instrumental method, the moisture determination has been performed by drying it in a closed furnace (confined space) with nitrogen atmosphere at 2.4 bar. Certainly, the two method differences, directly affect the difference in the obtained values.

Table 5. Moisture content in coal samples, determined by analytical and instrumental method

Sample label	W_a [%]	$W_{a,TGA}$ [%]	$W_{a,av}$ [%]	$W_{a,TGA,av}$ [%]
Kolubara 1	6.27	7.38	6.27	7.41
Kolubara 1	6.26	7.43		
Kolubara 2		6.70		6.74
Kolubara 2		6.78		
Kostolac 1	15.4	14.40	15.35	14.38
Kostolac 1	15.3	14.36		
Kostolac 2		6.65		6.69
Kostolac 2		6.73		
Brown coal	14.9	14.80	14.85	14.79
Brown coal	14.8	14.78		
Control sample	14.8	14.31	14.8	14.33
Control sample	14.8	14.35		

In the third step, the content of total carbon in the samples was determined using instrumental method with the aim of:

- comparisons of the order of magnitude of carbonate carbon in relation the total carbon content and
- determination, and confirmation of the coal category.

Based on the presented results of determining the total carbon, shown in tab. 6, it can be noticed that the content of total carbon for the samples Kolubara 1, Kolubara 2, Kostolac 1 and Kostolac 2, is in the range of about 35-40%, what confirms the these samples are lignites. For brown coal sample, its class was confirmed, based on the obtained value of total carbon of about 53%. Also for control sample, based on experimental tests, the content of total carbon of about 49-50% was obtained, which indicates that the sample is in the class of the brown coal. Several instrumental tests were repeated on the same sample and the mean value was calculated. The deviation of the individual measurement from the mean value was calculated. It is obvious that for each individual measurement deviation from mean value is below 1%, what confirms good repeatability of the measurement and method.

Determination of carbonate carbon and CO₂ content, in the samples, was performed by instrumental method, and the verification was performed using standard analytical gravimetric method. The obtained experimental data are shown in tab. 7. The mean values of the obtained results and the deviation between the two methods are shown in tab. 8. Insight into the obtained experimental data, shows a good agreement between the values of the carbonate carbon content. Exceptions are samples Kostolac 1 and Kostolac 2, for which deviation of the obtained experimental values is slightly higher. For Kostolac 1 sample, deviation is 29.4 %, while for Kostolac 2 sample the deviation is 14.3 %. The reason for such values deviation can be multiple, but two points are crucial. The first reason is that the methods are quite different from each other, as already mentioned when commenting on the moisture results. Another reason is that Kostolac samples probably contain certain mineral compounds that decompose at different higher/lower temperatures, which increased deviation. This assumption has been validated, since for pure CaCO₃ the deviation of the results from the stoichiometric content is less than 5.5%, what is an excellent agreement.

Table 6. Total carbon content values and its mean values in tested samples determined by instrumental method on the LECO CHN628 elementary analyzer

Sample label	W_c [%]	W_{av} [%]	Deviation [%]
Kolubara 1	37.920	38.2	0.73
	38.552		0.92
	38.073		0.33
Kolubara 2	38.445	38.5	0.14
	38.533		0.09
Kostolac 1	34.931	34.6	0.96
	34.269		0.96
Kostolac 2	38.099	38.1	0.00
	38.109		0.02
Brown coal	53.489	53.6	0.21
	53.760		0.30
	53.531		0.13
	53.759		0.30
Control sample	50.143	49.7	0.89
	49.992		0.59
	49.936		0.47
	49.336		0.73
	49.536		0.33
	49.453		0.50

Table 7. Carbonate carbon and CO₂ content in tested coal samples determined by analytical gravimetric and instrumental thermogravimetric method

Sample label	W_c [%]	W_{CO_2} [%]	$W_{c,TGA}$ [%]	$W_{CO_2,TGA}$ [%]
Kolubara 1	0.138	0.490	0.147	0.54
	0.144	0.528	0.156	0.57
Kolubara 2	0.128	0.468	0.164	0.60
	0.140	0.512	0.134	0.49
Kostolac 1	0.115	0.420	0.142	0.52
	0.102	0.374	0.139	0.51
Kostolac 2	0.176	0.646	0.172	0.63
	0.188	0.690	0.243	0.89
Brown coal	0.103	0.378	0.096	0.35
	0.110	0.402	0.104	0.38
Control sample	0.052	0.192	0.049	0.18
	0.039	0.144	0.041	0.15

The attention should also be paid to the order of magnitude for carbonate carbon (tabs. 7 and 8) in relation the values for total carbon obtained by the instrumental method, tab. 6. It is evident that bound carbon in carbonate mineral compounds is in negligibly small amounts relative to the total carbon content. This is established for Serbian lignites from open pit mines Kolubara and Kostolac and for two samples of coal of undetermined origin.

Table 8. Carbonate carbon and CO₂ content mean values for tested coal samples determined by analytical gravimetric and experimental thermogravimetric method

Sample label	$W_{av,C}$ [%]	$W_{av,C,TGA}$ [%]	W_{av,CO_2} [%]	$W_{av,CO_2,TGA}$ [%]	Deviation [%]
Kolubara 1	0.141	0.152	0.509	0.555	7.8
Kolubara 2	0.134	0.149	0.490	0.545	11.2
Kostolac 1	0.109	0.141	0.397	0.515	29.4
Kostolac 2	0.182	0.208	0.668	0.760	14.3
Brown coal	0.107	0.100	0.390	0.365	6.5
Control sample	0.046	0.045	0.168	0.165	2.2

Conclusions

In the paper carbonate carbon content in coal samples has been determined for four samples of lignite originating from Serbian open pit mines Kolubara and Kostolac, one sample of brown coal and one control sample of proficiency test scheme. The determination of the carbonate carbon content was performed using the analytical gravimetric method and a comparison was made with the tests performed by the instrumental method with a thermogravimetric analyzer. In addition, the determination of the moisture content in the samples and the determination of the total carbon content by the instrumental method using an elementary analyzer were performed. Based on the presented tests and obtained results, the following conclusions can be reported.

- Apparatus for analytical method has been successfully assembled and checked for tightness. The check was performed using CaCO₃.
- Based on the values of carbonate carbon content, obtained using both methods and their mutual comparison, the agreement is satisfactory, with the exception of samples Kostolac 1 and Kostolac 2.
- The reason for larger difference of the results from Kostolac open pit mine is primarily that the aforementioned methods whose results are compared differ substantially and it is likely that the samples that originate from Kostolac, contain certain mineral compounds that decompose at different higher/lower temperatures causing the deviation in question.
- Carbon bound in carbonate mineral compounds in the tested samples is present in negligibly small quantities in relation total carbon.

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Nomenclature

m_1 – test sample mass, (in eq. 3) [g]
 m_2 – increase in mass of absorption tube, (in eq. 3) [g]
 m_1 – mass of empty container with lid, (in eq. 1) [g]
 m_2 – mass of the container with lid and coal sample before drying, (in eq. 1) [g]

m_3 – mass of the container with lid and coal sample after drying, [g]
 W – moisture content, [%]
 W_C – carbonate carbon content, [%]
 W_{CO_2} – carbon-dioxide content, [%]
 W_a – analytical moisture content, [%]

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