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Microwave-Assisted Preparation of sinter samples for chemical composition analysis by IPC-OES technique

Abstract: The article discusses tests aimed to develop a method enabling the preparation of iron ore sinters applying microwave-assisted mineralisation and subsequently used in the ICP-OES technique-based chemical composition analysis. The tests involved the use of various mineralising mixtures, one of which was selected to prepare specimens of sinters used in the ICP-OES technique-based chemical composition analysis. The repeatability of the microwave-assisted mineralisation of sinters was investigated through the repeated mineralisation of the same sinter specimen performed under the same conditions. The test results revealed that the microwave-assisted mineralisation technique could be used routinely in ICP-OES technique-based sinter analytics. The preparation of the sinters performed using the above-named technique was less laborious and involved the use of small amounts of chemical reagents.

Key words: ICP-OES, microwave mineralization, iron ores, sinters

1. Introduction

Iron ore sinters belong to key feedstocks used in metallurgical processes. Because of their properties, the above-named sinters not only constitute an iron-bearing material (crucial as regards the quality of pig iron), but also support blast-furnace processes, ensuring the stable operation of the furnace as well as the easy exchange of heat and mass. The control of the chemical composition of sinters is an important factor responsible for quality in metallurgical processes. The preparation of sinter specimens for complex chemical composition analysis (regardless of a given technique applied in the analytical process) requires the proper handling of materials subjected to tests. Difficulties analysing the chemical composition of sinters results from their high resistance to the effect of chemical reagents, including mineral acids and their mixtures.

One of the techniques enabling the entire decomposition of materials being tested to the form of solution is microwave-assisted mineralisation. The first mentions in publications concerning the application of the microwave technique in the decomposition of specimens for chemical analysis appeared at the end of 1970s and the beginning of 1980s [1]. The process of decomposition in microwave-assisted mineralisation is performed in a reaction vessel, usually made of fluorinated polymer, such as polytetrafluoroethylene (PTFE) or perfluoroalkoxy polymer (PFA). After placing the specimen and providing necessary digestion reagents, the vessels is tightly closed and exposed to the effect of microwave energy. Initially, tests involving the decomposition of specimens were performed using household microwave cookers combined with improvised reaction gas exhaust systems [2]. Presently, mineralisation is performed using commercial mineralisers, which, depending on their design, enable the mineralisation of one or several specimens at the same time.

A significant advantage of microwave specimen preparation systems over conventional ones is the performance of the process in a dedicated closed reaction system. During microwave-assisted mineralisation, the specimens are exposed to three factors, i.e. microwave energy, high pressure and chemical reagents making up the reaction mixture. The heat generated by microwaves intensifies chemical reactions. The heating of the reaction mixture is accompanied by an increase in pressure in the reaction vessel by released gases. As a result, gaseous reagents cannot leave the reactor and the process of specimen decomposition can be performed at temperature significantly exceeding the boiling point of reagents used in the process. The above-presented conditions cannot be provided by the open system. The process of specimen decomposition is faster in the closed system. In addition, the use of the closed reaction environment reduces the risk of the loss of readily volatile analytes. The process of microwave-assisted mineralisation can involve both inorganic specimens and organic matrixes [3].

Presently, the microwave-assisted decomposition of specimens is a commonly used method when preparing solution specimens. Some of the major application areas of microwave-assisted mineralisation are environmental tests of waters [4], soils [5, 6] and air quality [7, 8]. Microwave-assisted chemical digestion is also applied in geology, where the analytical process requires the preparation of specimens resistant to such chemical agents as bottoms [9], ores [10, 11] and mineral deposits (being carriers of rare-earth elements) [12].

As microwave-assisted mineralisation makes it possible to decompose specimens having a rich organic matrix, it can be used in the petrochemical industry (where the fast determination of the wide spectrum of chemical elements is of great importance) [13] and in tests of polymer (internal) stabilisers, reducing flammability and having antistatic and sliding effects [14].

Microwave-assisted mineralisation is used in tests where specimens subjected to investigation are of biological origin [15]. A particular area where microwave-based

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Table 1. Operating parameters of the mineraliser used in the mineralisation of sinters

	Stage 1, preheating	**Stage 2, heating**	**Stage 3, cooling**
Duration [s]	300	600	600
Critical temperature range [°C]	240–250	310–300	-
Critical pressure range [MPa]	1.7–2.0	3.2–3.5	-
Generator power [%]	60	100	0

specimen preparation is applied is proteomics, i.e. the study of proteins for gene sequencing [16, 17] and biomarkers (used in the early detection of diseases) [18]. Medical research and research-related tests often necessitate the preparation of specimens in the form of solutions, which is of significant importance when testing the quality of medicines [19], their effect on the human body [20] and released metabolites [21]. Microwave-assisted mineralisation has also found application in tests of food [22], factors affecting food quality [23] and the usability of food industry waste [24].

In addition, microwave-assisted mineralisation can be used in the partial decomposition of specimens, i.e. where it is not necessary to entirely decompose a given specimen, but only to extract a single analyte or a group of compounds [26, 27]. The technique is also used in on-line technique-based analyses, where the material of a given specimen is subjected to mineralisation and, afterwards, transported (using a continuous stream) to devices recording analytical signals [27, 28].

Regardless of its application areas, the specific nature of microwave-assisted mineralisation enables the use of small volumes of reagents, making this method more environmentally friendly than traditional specimen decomposition methods [26].

One of the disadvantages of the microwave technique is the capacity of vessels and, the resultant possibility of mineralising specimens of weight not exceeding 500 mg. An element potentially affecting the efficiency of the process is the lack of the possibility of stirring the reaction solution, which leads to the non-uniform contact of the specimen with the reaction medium and reduces the efficiency of the mineralisation process. Another issue, inseparably connected with the microwave-assisted mineralisation technique is the risk of a sudden and uncontrollable pressure increase in the mineraliser, resulting in the burst of the reaction vessel. The above-named phenomenon is particularly common, where the reaction of the specimen with the medium releases excessive amounts of gaseous products [29].

Microwave-assisted mineralisation is predominantly used where it is necessary to prepare specimens, the entire decomposition of which in traditional open systems is impossible. At the same time, mineralisation process conditions reduce the risk of analyte loss. In spite of the vast applicability of microwave-assisted mineralisation, scientific reference publications contain very little information concerning the use of this technique in the analysis of sinters.

The tests discussed in the article aimed to identify the applicability of the microwave-assisted mineralisation of iron ore sinters in the ICP-OES technique-based analysis of chemical composition. The test results were compared with those obtained using the WD-XRF (reference) technique, applied to test sinter specimens after previously melting them to the form of pearl.

2. Research

2.1. Testing equipment

The tests concerning the applicability of microwave-assisted mineralisation in the preparation of sinter specimens involved the use of a Magnum II mineraliser (Ertec), i.e. a one-station pressure device. The process of microwave-assisted mineralisation was performed in a single vessel made of pressed Teflon and having a capacity of approximately 100 cm^3. All mineralisation tests were performed under the same process conditions (Tab. 1).

The determination of chemical composition was performed using a 5100 SVDV ICP-OES spectrometer (Agilent). During measurements, the spectrometer was equipped with a system enabling the transport of specimens (nebulizer resistant to the effect of hydrofluoric acid (i.e. “One Neb” nebuliser)), dual-pass plastic cloud chamber and a plasma torch with an injection tube made of aluminium oxide. The parameters of the ICP-OES spectrometer analytical software programme used in the determination of selected sinter constituents are presented in Table 2.

Table 2. Parameters of the ICP-OES spectrometer analytical software programme used in the determination of selected sinter constituents

Wavelength [nm]	Al 308.215
	Ca 317.933
	Fe 234.350
	K 766.491
	Mg 202.582
	Mn 293.305
	Na 588.995
	P 213.618
	Si 251.611
	Ti 334.941
	Zn 202.548
Plasma view	side
Plasma generator power [W]	1200
Argon flow rate [dm^3/min]	0.7
Argon plasma flow rate [dm^3/min]	12
Pump rotational rate [obr/min]	20
Cloud chamber	“One Neb”
Komora mgielna	dual-pass Teflon chamber

2.2. Specimen preparation

The efficient decomposition of specimens in the process of microwave-assisted mineralisation required the selection

Table 3. Composition of the reaction mixtures used in the sinter decomposition tests

Mixture designation	Composition of reaction mixture
M_1	7 cm^3 HCl + 1 cm^3 HF
M_2	8 cm^3 HCl + 2 cm^3 HF
M_3	6 cm^3 HNO_3 + 1 cm^3 HCl + 1 cm^3 HF
M_4	2 cm^3 HNO_3 + 6 cm^3 HCl + 1 cm^3 HF + 1 cm^3 H_2O_2
M_5	3 cm^3 HNO_3 + 3 cm^3 H_3PO_4 + 2 cm^3 HF
M_6	3 cm^3 HNO_3 + 1 cm^3 $HClO_4$ + 2 cm^3 HF
M_7	3 cm^3 H_2SO_4 + 2 cm^3 H_2O_2 + 3 cm^3 HF + 2 cm^3 H_2O
M_8	6 cm^3 HCl + 2 cm^3 HNO_3 + 3 cm^3 HF
M_9	3 cm^3 HNO_3 + 6 cm^3 HCl + 3 cm^3 HF
M_10	4 cm^3 HNO_3 + 4 cm^3 H_2SO_4 + 1 cm^3 HF

of the appropriate mixture of reagents, enabling the decomposition of the material, including constituents in the form of oxides (which, during the digestion process taking place in the open system, do not pass to the solution). The determination of mixture composition required the performance of a series of tests aimed at the mineralisation of sinter specimens using inorganic reagents mixed in various proportions (Tab. 3). All the tests were performed using the same sinter specimen and the same mineraliser operating conditions.

The initial criterion assessing the efficiency of the mineralisation process was based on the visual inspection of specimen digestion. All the solutions containing undissolved specimen leftovers (indicating incomplete decomposition) were rejected. At this stage of the tests, mixtures M_1, M_2 and M_9 were identified as the most efficient. Afterwards, the ICP-OES technique was applied to record the emission spectrum of solution constituents obtained after stirring mixtures M_1, M_2 and M_9. The comparison of the intensity of the emission spectrum of selected chemical elements revealed that the highest intensity of emitted radiation characteristic of chemical elements was identified in the solution obtained using the M_2: 8 cm^3 HCl + 2 cm^3 HF mixture.

2.3. Sinter preparation repeatability

The initial stage of the tests (during the preparation of the mineralisation process) involved the identification of several problems. As a result of the accumulation of electrostatic charges, during the weighing of specimen portions, the material (in the form of fine powder) tended to settle on the walls of the reaction vessel and could not be rinsed off the walls using the reaction mixture. Another problem was the formation of specimen agglomerates at the bottom of the reaction vessel after the addition of reagents. The chemical reagents could not efficiently penetrate the clusters of the specimen. The sedimentation of the specimens on the vessel walls and the formation of agglomerates restricted the contact of the mineralised material with the reaction mixture, which, in turn, resulted in incomplete decomposition. For this reason, during the weighing of the specimen as well as after providing chemical reagents it was necessary to ensure the uniform dispersion of the test material in the reaction mixture. Consequently, it was necessary to assess the repeatability of the procedure used for the preparation of sinter specimens by means of microwave-assisted mineralisation. To this end, it was necessary to perform (tenfold) the mineralisation of the same sinter specimen. The process was performed in 3 series, separated from each other by a minimum of 2 days. The criterion of mineralisation process repeatability adopted in the tests was the precision of results obtained (by one analyst) under the same conditions and using the same reagents within a short period of time. The mineralisation process precision was expressed using the coefficient of variability (CV) (Tab. 4). The values of CV did not exceed 5 %, which indicated satisfactory process repeatability.

3. Results and discussion

The results concerning the determination of selected sinter constituents obtained using the ICP-OES technique and the solutions prepared using the mineralisation technique were compared with those obtained using the WD-XRF (reference) technique (Tables 5 through 9), where sinter specimens were melted to the form of the pearl. The tests were performed using the specimens developed in the sinter plant of the Upper-Silesian Institute of Technology (of the Łukasiewicz Research Network).

The differences in terms of chemical analysis results between the ICP-OES technique and the reference technique (WD-XRF) were observed in relation to iron determination. Such an effect did not result directly from the specimen preparation (mineralisation) technique applied in the tests, but from the limited applicability of the ICP-OES technique in the determination of high concentrations of analytes. During the above-named determination, the recording of characteristic radiation intensity was performed for iron emission lines of lower intensity. Such an approach resulted from the necessity of reducing the analytical signal entering the spectrometer detector, which, after exceeding the threshold limit, stopped recording the emission spectrum of a given analyte subjected to determination. In spite of applying the analytical line of lower intensity, the difference in iron determination performed using both techniques did not exceed 2 %. The convergence of iron determination results with the results obtained using the reference technique (WD-XRF) demonstrated that microwave-assisted mineralisation could be applied during the preparation of sinter specimens even in the determination of the primary matrix constituent.

The comparison of the chemical composition analysis results obtained using the ICP-OES technique after specimen mineralisation with those obtained using the reference technique (WD-XRF) confirmed the applicability of microwave-assisted mineralisation in ICP-OES technique-based tests of sinter chemical composition.

Table 4. Coefficient of variability in the process repeatability tests

Wavelength [nm]	Mg 202.582	P 213.618	Mn 293.305	Ti 334.941	Na 588.995	K 766.491	Fe 234.350	Si 251.611	Al 308.215	Ca 317.933
Average concentration [% m/m]	0.38	0.013	0.027	0.025	0.042	0.035	64.18	6.58	0.40	2.77
CV [%]	2	3	3	5	4	4	4	4	2	3

Table 5. Comparison of results concerning specimen Sinter 1, obtained using the WD-XRF technique and the ICP-OES technique

Specimen: Sinter 1		
Constituents	**WD-XRF, melted specimen**	**ICP-OES, specimen after mineralisation**
Al_2O_3 [% m/m]	0.39 ± 0.03	0.41
CaO [% m/m]	2.71 ± 0.10	2.80
Fe [% m/m]	63.95 ± 0.44	61.18
K_2O [% m/m]	0.035 ± 0.005	0.036
MgO [% m/m]	0.36 ± 0.03	0.39
Mn [% m/m]	0.026 ± 0.002	0.027
Na_2O [% m/m]	0.042	0.043
P [% m/m]	0.014 ± 0.002	0.013
SiO_2 [% m/m]	6.12 ± 0.15	6.28
TiO_2 [% m/m]	0.026 ± 0.005	0.023

Table 6. Comparison of results concerning specimen Sinter 2, obtained using the WD-XRF technique and the ICP-OES technique

Specimen: Sinter 2		
Constituents	**WD-XRF, melted specimen**	**ICP-OES, specimen after mineralisation**
Al_2O_3 [% m/m]	0.70 ± 0.04	0.67
CaO [% m/m]	8.46 ± 0.25	8.50
Fe [% m/m]	57.65 ± 0.44	57.94
K_2O [% m/m]	0.023 ± 0.004	0.022
MgO [% m/m]	1.30 ± 0.08	1.22
Mn [% m/m]	0.062 ± 0.003	0.063
Na_2O [% m/m]	0.024	0.024
P [% m/m]	0.032 ± 0.003	0.033
SiO_2 [% m/m]	7.95 ± 0.19	8.01
TiO_2 [% m/m]	0.026 ± 0.005	0.024

Table 7. Comparison of results concerning specimen Sinter 3, obtained using the WD-XRF technique and the ICP-OES technique

Specimen: Sinter 3		
Constituents	**WD-XRF, melted specimen**	**ICP-OES, specimen after mineralisation**
Al_2O_3 [% m/m]	0.30 ± 0.03	0.27
CaO [% m/m]	2.83 ± 0.10	2.79
Fe [% m/m]	65.00 ± 0.44	64.12
K_2O [% m/m]	0.049 ± 0.006	0.052
MgO [% m/m]	0.34 ± 0.03	0.29
Mn [% m/m]	0.022 ± 0.002	0.025
Na_2O [% m/m]	0.070	0.065
P [% m/m]	0.013 ± 0.002	0.010
SiO_2 [% m/m]	5.15 ± 0.13	5.14
TiO_2 [% m/m]	0.033 ± 0.005	0.032

4. Summary

At the initial stage of the tests it was possible to observe that the preparation of specimens for microwave-assisted mineralisation significantly affected the efficiency of the digestion process. Key aspects included the proper placement of the specimen in the reaction vessel and providing the uniform access to the reaction mixture. The search for a mixture enabling the mineralisation of sinter specimens involved the testing of numerous mixtures of inorganic acids. The tests revealed that the process of microwave-assisted mineralisation in an aqueous environment was more selective. For this reason, the composition of the reaction mixture had to be adjusted strictly in relation to the mineralisation of a given specimen. The mixture qualified as being optimum for the digestion of the test sinters was 8 cm^3 HCl + 2 cm^3 HF. The assessment of microwave-assisted mineralisation repeatability necessitated the per-

Table 8. Comparison of results concerning specimen Sinter 4, obtained using the WD-XRF technique and the ICP-OES technique

Specimen: Sinter 4		
Constituents	**WD-XRF, melted specimen**	**ICP-OES, specimen after mineralisation**
Al_2O_3 [% m/m]	0.71 ± 0.04	0.69
CaO [% m/m]	7.78 ± 0.23	7.71
Fe [% m/m]	58.35 ± 0.44	59.12
K_2O [% m/m]	0.010 ± 0.004	0.010
MgO [% m/m]	1.17 ± 0.07	1.14
Mn [% m/m]	0.039 ± 0.003	0.042
Na_2O [% m/m]	0.043	0.041
P [% m/m]	0.028 ± 0.003	0.033
SiO_2 [% m/m]	7.93 ± 0.19	8.12
TiO_2 [% m/m]	0.032 ± 0.005	0.033

Table 9. Comparison of results concerning specimen Sinter 5, obtained using the WD-XRF technique and the ICP-OES technique

Specimen: Sinter 5		
Constituents	**WD-XRF, melted specimen**	**ICP-OES, specimen after mineralisation**
Al_2O_3 [% m/m]	0.87 ± 0.04	0.85
CaO [% m/m]	9.89 ± 0.28	9.73
Fe [% m/m]	56.68 ± 0.43	57.15
K_2O [% m/m]	0.024 ± 0.005	0.024
MgO [% m/m]	1.35 ± 0.08	1.35
Mn [% m/m]	0.036 ± 0.003	0.041
Na_2O [% m/m]	0.028	0.027
P [% m/m]	0.035 ± 0.003	0.033
SiO_2 [% m/m]	7.72 ± 0.18	7.65
TiO_2 [% m/m]	0.032 ± 0.005	0.031

formance of the multiple decomposition of the same sinter specimen under the same conditions. The repeatability of results was based on the determination of the coefficient of variability (CV). In relation to the sinters subjected to determination, the value of CV did not exceed 5 %, which indicated high process repeatability. The comparison of the test results concerning the chemical composition of the test sinters performed using the ICP-OES and WD-XRF techniques revealed the high conformity of both methods, which, in turn, confirmed the applicability of microwave-assisted mineralisation as a method enabling the preparation of sinter specimen for ICP-OES technique-based tests of chemical composition.

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