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TESTING OF GEOPOLYMER RAW MATERIALS: VALIDATION OF METHODS AND PRACTICAL ASPECTS

The most used raw materials to produce geopolymers are metakaolin, fly ash, mine tailings, and granulated blast furnace slag. The geochemical composition of the starting materials is decisive for the structure and physical properties of the newly synthesized geopolymer. The present research is aimed at validating methods to determine pH, electrical conductivity of initial raw materials for the preparation of geopolymers. Methods trueness and precision were estimated by bias, z-score and repeatability determination. The results from certified reference material clay soil for pH and EC CRM498-100G, Lot LRAC5544 tests showed that the bias was 0.18% for pH and –1.7% for EC. The repeatability was 0.18% and 2.38% for pH and EC, respectively. The z-score was below 2 and the analytical behavior of both studied methods was evaluated as satisfactory.

The validated procedures were applied to mine tailings and coal combustion by-products from four sources from Bulgaria, Romania, and Portugal. The aqueous slurry of industrial wastes was with pH 6-12, all studied fly ashes contained high concentration of components ionized in water solution. The results showed that the studied raw materials could be used as precursors for geopolymer.

Keyword: Geopolymers; Raw Materials; Validation; pH; Electrical Conductivity

1. Introduction

In recent years, worldwide, overview studies on geopolymers have increased, which confirms the interest of researchers in the subject. Geopolymers are class of tree-dimensionally networked aluminosilicate materials like zeolites first developed and investigated by Joseph Davidovich [1]. J. Davidovits reported that geopolymers, produced for industrial application immobilised hazardous elemental wastes within the geopolymeric matrix, as well as acting as a binder to convert semi-solid waste into an adhesive solid. Hazardous elements present in waste materials are «locked» into the three-dimensional framework of the geopolymeric matrix wastes [1].

The starting material for geopolimerization must be rich in silicon (Si) and aluminum (Al). These can be natural minerals such as metakaoline [2], kaolinite [3,7] or by-products such as fly ash [1,3,7], blast furnace slag [1,4,5], rice husk ash [4], etc.

Despite the growing interest in obtaining geopolymers from industrial waste, most research is related to the study of curing temperature, activator concentration, and nature of alkaline activator on the mechanical properties of the final product. In article [6] has been studied the influence of a series of factors as specific surface of the slag, curing temperature, activator concentration, and nature of alkaline activator on the mechanical strengths of alkaline-activated slag cement mortars.

The effect of composition and temperature on the properties as well as the curing regime of fly ash- and kaolinite-based geopolymers has been investigated in research [7]. In [8] the influence of curing temperature on the phase transformation, mechanical properties, and microstructure of the kaolin-ground granulated blast furnace slag (GGBS) geopolymer has been investigated.

The influence of silica fume on the thermal performance of fly ash geopolymers have been studied [9].

The latest research is focused on the rapidly developing geopolymer-reinforced fibres. The review article [9] shown material and geometrical properties, numerical simulation, and the effect of fibres on the geopolymers.

Another direction for geopolymer industrial utilisation is focused on geopolymer-based adsorbents of heavy metal. It was shown that geopolymer-based adsorbent, produced from raw materials such as fly ash, metakaolin, slag and kaolin [10] possesses exceptional pollutant removal properties.

The increase in the accumulation of tailings from the mining industry creates environmental and health problems. On the other hand, due to the limited sources and availability of metakaolin

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and reactive fly ash, most research has focused on mine tailings. In the review article [11], it was shown that the deposition of tailings has instantaneous impacts not only on the ecosystem, but also raises concern regarding the possibility of soil and water contamination with potentially toxic elements (PTE), such as arsenic (As), cadmium (Cd), and lead (Pb) [12].

The most important factor controlling the compressive strength of fly ash-based geopolymers is the pH of the starting material [13]. When cement is used as an additive in the geopolymer matrix, the compressive strength increases almost exponentially with increasing pH. In an article [14] pH and conductivity were determined by weighing 20 g of air-dried and homogenized samples and adding 20 ml of distilled water until a slurry was obtained. After mixing for 5s, the suspension was allowed to stand for 10 min. The pH/EC electrodes were then inserted into the suspension, and, after gentle rotation, the pH and EC were measured until a constant value was obtained.

The higher alkali content was found to promote solid dissolution but also caused aluminosilicate gel precipitation at very early stages thus resulting in a lower compressive strength [15].

Different authors use different approaches to determine acidity (pH) of mine tailings and obtained data cannot be compared. In resent research [16] mine tailings pH values have been measured at solid-to-water ratio = 1:2,5.

One of the main requirements of ISO/IEC 17025:2017 [17] is that the methods developed in the laboratory are validated. The goal of validation is to ensure that the analytical method is fit-to-purpose in terms of the uncertainty of the results, the limit of detection, repeatability, selectivity of the method, linearity, and sensitivity to the matrix of the test object. Proper method validation ensures that valid and reliable results are obtained.

The pH and electrical conductivity (EC) determination of soil have been analyzed through a few international standards [18-20]. In standard [18] regarding soils and industrial waste a ratio of solid material: water –1:5 (at least 5 mL test sample in a measuring spoon and 25 mL water) and a stirring time of 1 h (for pH) and 30 min (20 g of material and 100 ml of water) for conductivity [19] were used.

According to [20] the solid material: distilled water ratio is 1:1 (20 g of material and 20 mL of distilled water), followed by vigorous stirring for 5 min and a 1-hour dwell for phase separation, or filtration or centrifugation.

Analytical method validation is conducted to achieve quality assurance and quality control of chemical analysis with the goal of eliminating analytical bias [21]. Bias is a systematic error defined as the difference between the measurement result and its unknown 'true value' [22]. Data accuracy covers the concepts of data precision and data trueness [23]. Trueness can be quantified as bias, the systematic error. A reference material is defined as "a material of whose property values are sufficiently homogeneous and well established to be used for the calibration of a device, the assessment of a measurement method, or for assessing values to materials" and are available as certified or internal reference material [24].

Methods validation must be carried out on enough representative matrices of the product family [25-27]. Due to the lack of a certified reference material (CRM) based on tailings or fly ash, the methods were validated with CRM of soil. Analyzing CRM of soils is usually a procedure for estimating the analytical behavior of the applied method for testing mine tailings, however the discrepancy of the matrix composition or analytes content, as well as their state and speciation in the sample, doesn't allow the method to be fully studied.

The objectives of this study are to validate the methodology for measuring pH and electrical conductivity of mine tailings and fly ashes.

2. Experiment

2.1. Sample description

Four copper mine tailings and four ashes from thermal power plants (TPP) from Bulgaria, Romania and Portugal were studied. The sampling was made by the corresponding company applying their in-laboratory sampling plan. The samples were air-dried, homogenized without particle size reduction, and stored at room temperature. The samples description is presented in TABLE 1. A fraction below <200 μm was subjected to further analysis.

2.2. Method Validation pH and electrical conductivity

CRM clay soil for pH and EC: CRM498-100G, Lot LRAC5544, Expiration date April 2027 was used in the validation study.

The pH and EC were measured of 20 g of air-dried and homogenized geopolymer's raw material and CRM with 20 mL of distillate water in a baker [20]. The mixture was shaken for 5 min and pH was measured in the supernatant after 60 min. A HI5521-02 Laboratory Research Grade Benchtop pH/mV and EC/TDS/Salinity/Resistivity Meter was used for measurements. The calibration is done with buffer solution pH – 4.00; pH – 7.00; pH – 10.00 and a Conductivity Standard HI7030L 12880 μS/cm.

Each measurement was done 6 replicates, and the mean value was presented with specified uncertainty [28].

2.3. Mathematical approaches

Regression analysis, average, standard deviation (SD), reversed standard deviation (RSD, %) of the data were determined using Microsoft Office Excel 365 (New York, NY, USA).

In Eurachem Guide 2014 [29] have been use the three related performance characteristics trueness, precision, and uncertainty to describe the quality of results obtained with a method. However, scientists frequently use different concepts,

such as types of error (random, systematic), accuracy (trueness and precision) and uncertainty. Some of these concepts have a qualitative meaning and some are quantitative. Over the years, terms as well as definitions have changed, and new terms have been introduced.

The uncertainty was estimated according to the procedure recommended by EURACHEM/CITAC Guide [29]. In estimating the overall uncertainty, it may be necessary to take each source of uncertainty and treat it separately to obtain the contribution. Each of the separate contributions to uncertainty is referred to as an uncertainty component. For a measurement result y, combined standard uncertainty, denoted by $uc(y)$, is an estimated standard deviation equal to the positive square root of the total variance obtained by combining all the uncertainty components, however, evaluated, using the law of propagation of uncertainty (see Eq. (1)). For most purposes in analytical chemistry, an expanded uncertainty U, has been used. The expanded uncertainty provides an interval within which the value of the measurand is believed to lie with a higher level of confidence.

Three sources of uncertainty were included in the uncertainty budget.

$$u_c(x) = \sqrt{u(x_1)^2 + u(x_2)^2 + 2u(x_3)^2} \quad (1)$$

$u(x_1)$ – uncertainty from repeatability
$u(x_2)$ – uncertainty from CRM
$u(x_3)$ – uncertainty from additive error when weighing with the analytical balance.

The expanded uncertainty calculated using a coverage factor of 2 ($k = 2$) and 95% confidence level, was presented with: $U = 2$. uc, where uc was a combined uncertainty.

The Precision was given in term of repeatability. The tests were made according to the recommendation of Eurachem Guide 2014 [29]. The repeatability test was made for a short period, by the same user with the same instrument. Measurements were made in 6 replicates.

The trueness of the method was estimated by analysis of certified reference material in 6 replicates.

The bias was calculated (Eq. (3) [29]).

$$bias = \frac{X_{lab} - X_{ref}}{X_{ref}} 100, \% \quad (3)$$

The relative recovery in per cent is also calculated:

$R = \frac{X_{lab}}{X_{ref}} 100, \%$ [29].

Zeta-score was used as an estimate of the method trueness and performance indicator.

Zeta-score was calculated as [30]:

$$Z = \frac{X_{lab} - X_{ref}}{SD_{ref}} \quad (2)$$

Were X_{lab} is the mean value obtained in the laboratory; X_{ref} is the actual value of CRM; SD_{ref} – CRM Standart deviation.

3. Results and discussion

Four copper mine tailings and four ashes from thermal power plants (TPP) were studied. The sampling was made by the corresponding company applying their in-laboratory sampling plan. The samples were air-dried, homogenized without particle size reduction, and stored at room temperature. The samples description is presented in TABLE 1. A fraction below <200 μm was subjected to further analysis.

TABLE 1

Sample description

Sample	Description	Waste type
MT1	Copper mine tailing from Ellatzite, Bulgaria (Active tailing dump)	Mine tailing
MT2	Copper mine tailing from Assarel, Bulgaria (Active tailing dump)	
MT3	Mine tailing from Alum s.a Tulcea, Romania (red mud)	
MT4	Mine tailing from Pesqueira, Portugal	
FA1	Fly ash from TPP "Bobov Dol" thermal power station, Bulgaria	Fly ash
FA2	Mixed ash (fly ash, bottom ash, and coal dust) from TPP "Maritza East 2", Bulgaria,	Mixed ash
FA3	TPP ash dump from Romania	Fly ash
FA2	Coal ash from Prego TPP, Portugal	

For validation of method performance and uncertainty calculations of measurement of pH, EC six parallel samples of CRM were prepared, according [20]. From these data, the trueness (z-score), repeatability, recovery and relative bias of the method were calculated. The results are presented in TABLE 2.

TABLE 2

Analysis of CRM clay soil CRM498-100G, Lot LRAC5544

		pH	Electroconductivity at 25°C, μS/cm
CRM certificate data	Certified Value ± Expanded uncertainty	7.45 ± 0.05	2467 ± 128
	Confidence Interval	7.03 to 7.89	1730 to 3210
	Standard Deviation	0.143	247
Analyzed CRM (6 rep.)	Value ± Expanded uncertainty	7.46 ± 0.06	2429 ± 150
	Standard Deviation	0.0121	58
	Rel. bias, %	0.18	–1.70
	z-score	0.20	0.33
	Relative recovery %	100.13	98.30
	Repeatability (RSD, %)	0.16	2.38

The blank sample was with EC 1 μS/cm.

Assessment of z-scores is based on the following criteria:

- |z-score| ≤ 2.0 is regarded as satisfactory.
- 2.0 < |z-score| < 3.0 is regarded as questionable ('warning signal').
- |z-score| ≥ 3.0 is regarded as unsatisfactory ('action signal').

This is based on the concept that normally distributed analytical results lie within two standard deviations with a probability of 95%, and within three standard deviations with a probability of 99.7%.

The METHOD 9045D SOIL AND WASTE pH was validated and has demonstrated that its performance characteristics are adequate for use for a particular purpose.

The obtained zeta-score was less than 2.0 and the method performance was considered as "satisfactory" [31]. The method was applied to determination of pH and EC of four mine tailing and four fly ashes from Bulgaria, Romania, and Portugal (TABLE 3).

TABLE 3

Physicochemical characteristics of geopolymer's raw materials

Waste type	Sample	pH, 23°C	EC, mS/cm 23°C
Mine tailing	MT1	7.89±0.16	0.896±0.131
	MT2	8.47±0.28	1.951±0.326
	MT3	11.61±0.08	2.877±0.085
	MT4	5.98±0.33	1.184±0.160
Fly ash	FA1	12.84±0.24	2.508±0.112
	FA2	8.47±0.53	2.343±0.168
	FA3	13.17±0.14	4.868±0.092
	FA4	11.85±0.18	2.437 ±0.299

The studied mine tailing samples were alkaline with pH varied between 5.98 and 11.60. The obtained results are like those obtained in our previous studies conducted according to the conditions of other authors who studied mine tailings. Copper tailings leachates (MT1 and MT2) were typically alkaline pH ≈ 8-9, as it was reported for copper tailings from other sources [14]. The high alkalinity of the leachate pH 10-12 could partially explain the relatively high concentrations of CaO. The gold tailings had acidic pH 2-3 [32]. The low pH in goal tailings had been explained to the content of sulphides (pyrite (FeS2), sphalerite (ZnS), galena (PbS) [33].

According to literature data [14,34] high pH of the waste leads to faster fixation of the geopolymer, which can lead to technological problems in the synthesis process.

The electroconductivity of aqueous leachates was used to estimate the total concentration of dissolved charged components of the studied tailings. The highest EC and correspondingly, highest concentration of dissolved charged components was observed in fly ash sample indicating the high reactivity of the raw material.

4. Conclusions

The validation of analytical procedure for pH and electrical conductivity of aqueous slurry was done by applying a soil certified reference material. The results from CRM tests showed that the bias was 0.18% for pH and –1.7% for EC. The repeatability of CRM tests was 0.18% and 2.38% for pH and EC, respectively. The z-score was below 2 and the analytical behavior of both studied methods was evaluated as satisfactory.

The validated procedures were applied to mine tailings and coal combustion by-products from four sources from Bulgaria, Romania, and Portugal. The aqueous slurry of studied fly ashes was alkaline with pH from 8.47±0.53 for mixed ash (FA2) to 13.17±0.14 for TPP ash dump from Romania (FA3). The aqueous slurry of mine tailing was also alkaline (with pH 7.89±0.16 for MT1; 8.47±0.28 for MT2; 11.61±0.08 for MT3). The mine tailing from Pesqueira, Portugal (MT4) was with lowest pH 5.98±0.33.

All studied fly ashes and mine tailing from Alum s.a Tulcea, Romania (red mud) contained high concentration of components ionized in water solution. From studied materials, the highest value of EC has TPP ash dump from Romania (FA3) 4.868±0.092 mS/cm. The results showed that the studied mine tailings and fly ashes could be used as useful precursors for geopolymer.

5. Future Scope of Research

Validated methods for measuring the pH and electrical conductivity of soils and industrial wastes (mine tailings and fly ash) could be used by researchers in the characterization of feedstocks for geopolymer production to ensure reliability when comparing results.

The research raises the question of the need to prepare a certified reference material- mine tailings and flay ash, to be used in this direction, not only when measuring pH and conductivity, but also when determining other main characteristics of the starting raw materials, such as the BCR and other sequential extractions.

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