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Methodology for Testing the Uniformity of the Composition of a Batch of Polymer Materials on the Example of Sbr Rubber Granulates in the Aspect of Potential Applications

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ABSTRACT

This publication presents a comprehensive method for assessing the homogeneity of the composition of a large batch of polymer materials, with a mass of 1.5 tons, consisting of 60 transport packages, using the example of rubber granulate from recycled car tires. The presented method combines statistical analysis tools used to determine the samples number to be taken from the batch and the pyrolytic thermal desorption technique coupled with gas chromatography-mass spectrometry (Py/TD-GC-MS), used for material identification. Directions for improving the method for the quantitative identification of batch components are also indicated. The developed method can be applied to assess the purity of batches of recycled rubber granulate used in the construction of sports field surfaces and to determine the content of substances harmful to the environment and human health, such as polycyclic aromatic hydrocarbons. The direction for improving the developed spot-check control method using an alternative approach is to supplement it with a quantitative assessment of batch parameters in relation to the threshold values, such as tolerance limits.

Keywords: polymer materials, recycling, batch composition homogeneity, rubber granulate, styrene-butadiene rubber, physicomechanical properties.

INTRODUCTION

Every year, over 26 million tons of polymer waste are generated in the European Union, of which 31% is sent to landfills and 39% is incinerated, while the recycling rate does not exceed 30%. This persistently low recycling rate for polymer materials contributes to increasing environmental pollution, particularly in oceans and seas, where between 5 and 13 million tons of plastics are deposited annually worldwide, including up to 500,000 tons in the European Union [1]. To reduce ecosystem pollution from polymer materials, the European Commission has introduced a circular economy strategy for plastics aimed at increasing the recycled content in products while ensuring their efficiency and safety [2].

Material composition is the key parameter determining the direction of using the polymer materials for recycling. Identifying the type of plastic or rubber is a complex task, especially when there is a need for rapid identification of large quantities of samples with heterogeneous composition. Full analysis of a given polymer material requires significant time and advanced analytical equipment. It is necessary to first separate the plastic into its components, such as polymer, fillers, plasticizers, pigments, and others.

For the identification of the basic component of plastic, which is the polymer, differential

scanning calorimetry (DSC), thermogravimetry (TGA), and Fourier-transform infrared spectroscopy (FTIR) are used. The type of plastic component is determined using DSC and TGA methods based on the thermal profile or decomposition temperature obtained during the sample analysis, which are characteristic for each polymer [3]. In the case of the FTIR method, polymer identification is based on comparing the infrared absorption spectrum (IR) obtained for the tested sample with reference spectra of polymers [4].

For a detailed analysis of plastics comprising complex mixtures of polymers and additives, the pyrolytic thermal desorption method coupled with gas chromatography and mass spectrometry detection (Py/TD-GC-MS) is used. In the Py/TD-GC-MS method, the identification of plastic components is based on the analysis of mass spectra obtained for the thermal decomposition products of the sample during pyrolysis. The mass spectra are compared with databases containing reference spectra of specific polymer decomposition products [5]. This method features high analytical resolution, allowing the identification of even small amounts of plastic components [6]. However, the result of a single analysis cannot be directly related to the entire batch due to the small mass of the analytical sample compared to the batch mass. Thus, the results may be burdened with errors when assessing the batch of recycled materials.

Considering that the recycling process produces batches of polymer materials divided and packed into transport containers that can hold quantities ranging from 25 to 1000 kg, it is necessary to complement batch composition test with an assessment of their homogeneity. This assessment should include both the composition in each container and its comparison with the composition of other containers to confirm that the material composition of the entire batch is uniform and consistent with the recycler's declaration. This will ensure and maintain the high quality of final products made from recycled polymer materials, such as rubber granulate used for sports field construction and building materials [7,8].

The process of assessing the homogeneity of the composition of bulk polymer material batches requires selecting the proper sampling and sample preparation methods for chemical analysis, considering the necessary number of samples to interpret the results from various parts of the batch using statistical analysis tools. Essential aspect of sample preparation, regarding the required mass of the sample for chemical analysis, is the minimization of the laboratory sample mass in such a way to ensure the representativeness of the material taken for testing.

The testing methods used to identify types of plastics, including standardized ones [9, 10], do not consider assessing the composition homogeneity of the of bulk material batches and need to be supplemented in this regard. Statistical quality control (SQC) methods used in assessing the compliance of the product properties with technical requirements [11] can be applied. The interpretation of test results using SQC methods with alternative and numerical evaluation, employing statistical analysis tools, allows for reducing the scope of costly and time-consuming inspection of products to tests of a statistically determined sample size. Based on such test results, a decision is made to accept or reject the batch, keeping the acceptable quality level (AQL), which defines the threshold value of the satisfactory percentage of non-compliant parts. However, ensuring the reliability of batch composition assessment results requires proper sample collection for testing, following the statistical principles [11]. The sample must be taken from a batch produced in the same conditions, from the same raw materials, using the same technologies, machines, and equipment, stored under the same conditions. Sampling to ensure representativeness must be performed in a way that considers the required sample size for the method used and the required AQL [12,13].

A separate problem is the influence of regranulate additives on the operating properties closely related to the application area. There are known works on the introduction of regranulate to pure material, but the physicomechanical properties change. The decrease in properties depends on the method of obtaining the regranulate, its content and the degree of its contamination [14, 15]. There are known works on various modifications of the properties of SBR rubber, e.g. by using oil plasticizers, which reduce the viscosity but also influence the mechanical properties [16]. Often, waste SBR is used to produce blends, e.g. with recycled acrylonitrile-butadiene rubber filled with carbon black and silica, treated with and without a silane coupling agent. The studies conducted have shown that the use of silane improved the strength properties of the prepared materials. However, they were dependent on the degree of cross-linking and the silica content [17]. An important factor determining

the physicomechanical properties of a material is the manufacturing technology.

This publication presents results of the test conducted at the KOMAG Institute on the homogeneity assessment of polymer material batches using the developed method, combining the tools used in SQC with chemical analyses of material composition. It also presents the sample collection process from bulk polymer material batches and their preparation for material identification testing.

Figure 1. SBR rubber granulate for homogeneity composition test [18]

COMPOSITION AND PURITY TESTS

For testing the homogeneity of the polymer materials batch composition, a sample from the batch of rubber granulate from the recycled car tires was selected.

This batch consisted of 60 transport containers, representative in size for delivery of this type of raw material. Each container contained rubber granulate weighing 25 kg with a grain size of 0.5–2 mm, declared by the manufacturer to be styrene-butadiene rubber (SBR) – see Figure 1 [18].

The transport containers were numbered from 1 to 60. To confirm that the tested batch does not contain more than 1% by mass of granulate other than SBR, an acceptable quality level (AQL) of 1% was adopted for the batch composition tests. Assuming a stable recycling process, a standard control was used in testing, rejecting other control levels including strict one, recommended when the quality of at most 2 out of 5 consecutive batches deviates from the required standards, or reduced, applied when the quality of 10 consecutive batches meets the requirements [11].

The control plan, including the number of laboratory samples and the acceptance (Ac) and rejection (Re) numbers, was determined based on Table 1. For the established acceptable quality level of 1%, a batch size of 60 packages, and the normal inspection level, the number of laboratory samples was determined to be 13 (denoted by letter E), with criteria for assessing the results of composition tests conducted for the batch, including acceptance number Ac = 0 and rejection number Re = 1.

Transport packages for laboratory sampling were selected randomly. To select random sample numbers for identifying the type of polymer material, using the function RAND.BETWEEN (lower; upper) in Microsoft Excel 2010, n = 13 random package numbers were generated from 1 to 60. The random numbers were: 5, 10, 15, 23, 25,

Table 1. Normal inspection plans

Batch Size	Sample Size Code Letter	Laboratory Sample Size	Acceptable Quality Level [%]																					
			0.10		0.15		0.25		0.40		0.65		1.0		1.5		2.5		4.0		6.5		10	
			Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re	Ac	Re
2 to 8	A	2	↓		↓		↓		↓		↓		↓		↓		↓		↓		0	1	↑	
9 to 15	B	3	↓		↓		↓		↓		↓		↓		↓		↓		0	1	↑		↓	
16 to 25	C	5	↓		↓		↓		↓		↓		↓		↓		0	1	↑		↓		1	2
26 to 50	D	8	↓		↓		↓		↓		↓		↓		0	1	↑		↓		1	2	2	3
51 to 90	E	13	↓		↓		↓		↓		↓		0	1	↑		↓		1	2	2	3	3	4
91 to 150	F	20	↓		↓		↓		↓		0	1	↑		↓		1	2	2	3	3	4	5	6
151 to 280	G	32	↓		↓		↓		0	1	↑		↓		1	2	2	3	3	4	5	6	7	8
281 to 500	H	50	↓		↓		0	1	↑		↓		1	2	2	3	3	4	5	6	7	8	10	11
501 to 1200	J	80	↓		0	1	↑		↓		1	2	2	3	3	4	5	6	7	8	10	11	14	15
1201 to 3200	K	125	0	1	↑		↓		1	2	2	3	3	4	5	6	7	8	10	11	14	15	21	22
3201 to 10000	L	200	↑		↓		1	2	2	3	3	4	5	6	7	8	10	11	14	15	21	22	↑	

Note: Developed based on [11]

- ↓ Use the first plan below the arrow. If the number of laboratory samples is equal to or greater than the lot size, 100% inspection should be applied;
- ↑ Use the first plan above the arrow;
- Ac Acceptance number;
- Re Rejection number.

31, 37, 38, 47, 49, 52, 53, 54. From the transport packages marked with the aforementioned numbers, at 3 measurement points located at 1/3, 1/2, and 2/3 of the package height, 3 single samples of granulate were taken using a sampler for loose materials compliant with ISTA guidelines and meeting the requirements of PN-EN ISO-24333 [19,20]. The mass of each collected sample was approximately 50 g. Each sample was averaged through homogenization to create a laboratory sample. Then, each of the 13 laboratory samples was quartered to obtain 2 analytical samples.

Composition of the analytical samples was determined using the method of pyrolytic thermal desorption coupled with gas chromatography-mass spectrometry (Py/TD-GC-MS). At the first stage, chromatographic analyses of the reference material of styrene-butadiene rubber (SBR) were carried out. In the obtained chromatographic analysis of the reference material, characteristic peaks of pyrolytic decomposition products of SBR were identified (Figure 2).

For the peaks identified in the chromatogram of the reference material, mass spectra of pyrolytic decomposition products of SBR were prepared – reference spectra (Table 2 and Figure 3a–i).

At the second stage, chromatographic analyses of each analytical sample were conducted. Each analysis was performed for two parallel analytical samples. The results of the analyses were compared with the chromatogram of the reference material SBR and the reference mass spectra presented in Figure 3.

The results of chromatographic analyses, determining the type of material, were evaluated in light of the adopted criteria for batch uniformity

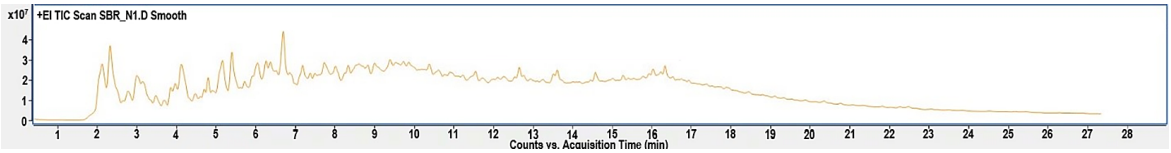
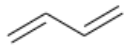
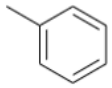
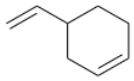
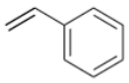
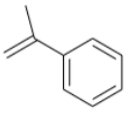
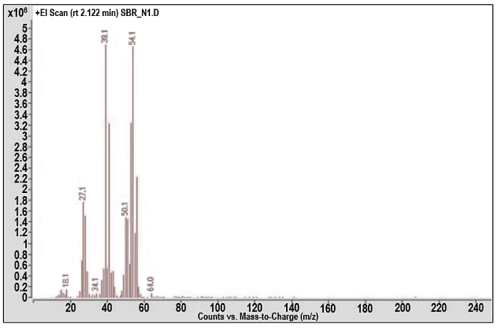


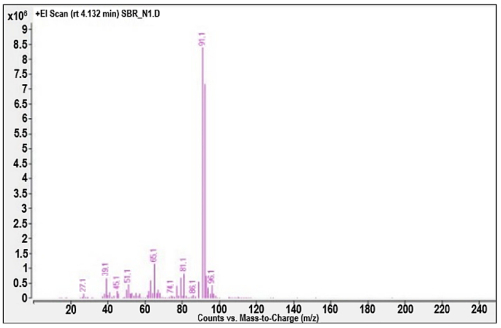
Figure 2. Chromatogram of the reference material of styrene-butadiene rubber (SBR) – TIC (total ion chromatogram)

Table 2. Characteristics of pyrolytic decomposition products of the reference material SBR [5, 18]

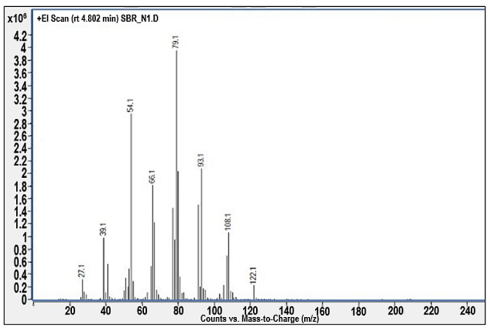
Pyrolytic decomposition product	Molecular weight [g/mol]	Fragmentary ions [m/z]	Structure
1,3-butadiene (B)	54	39, 29, 54	
Toluene (TO)	92	91, 29, 39, 51, 65	
4-vinylcyclohexene (D/B dimer)	108	54, 29, 39, 66, 79, 93, 108	
Styrene (S)	104	104, 39, 51, 63, 78, 103	
α-methylstyrene	118	118, 39, 51, 63, 78, 103, 117	
C12H18 (B trimer)	162	91, 32, 39, 55, 65, 77, 115, 129, 146	–
C12H18 (B trimer)	162	93, 29, 41, 53, 67, 79, 106, 119, 133, 147, 162	–
C12H14 (SB hybrid dimer)	158	104, 29, 39, 51,65, 78, 91, 128, 143, 158, 172	–
C16H12 (SBB hybrid trimer)	204	104, 41, 53, 65, 79, 91, 129, 143, 169, 183, 212	–



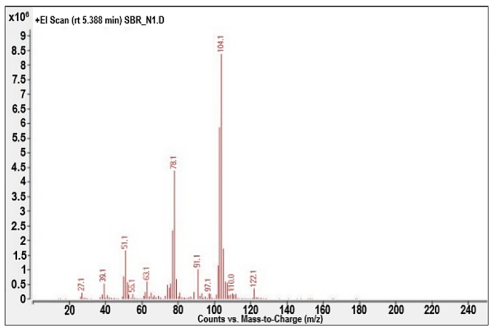
a) 1,3-butadiene (B)



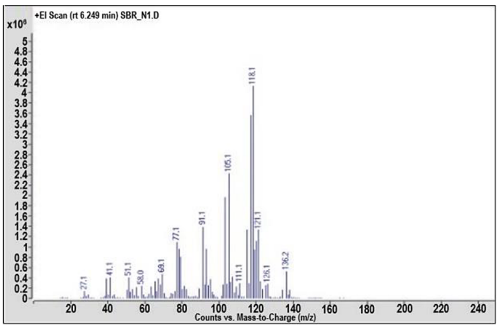
b) Toluene (TO)



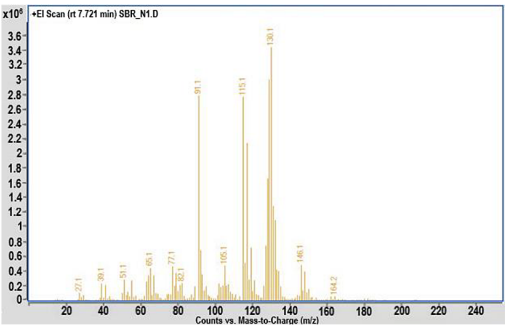
c) 4-vinylcyclohexene (D/B dimer)



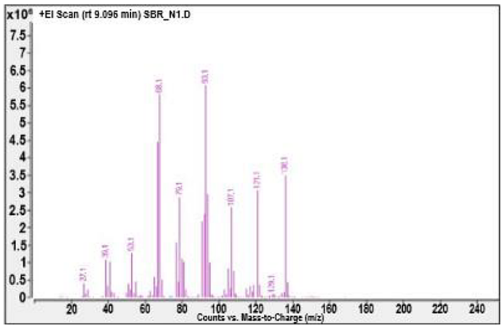
d) Styrene (S)



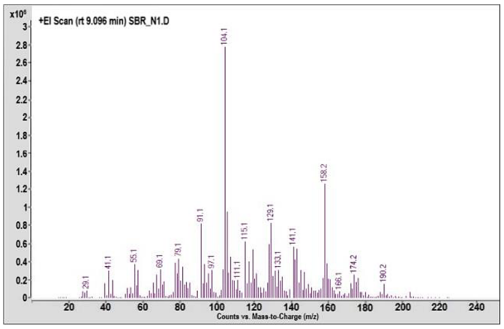
e) α-methylstyrene



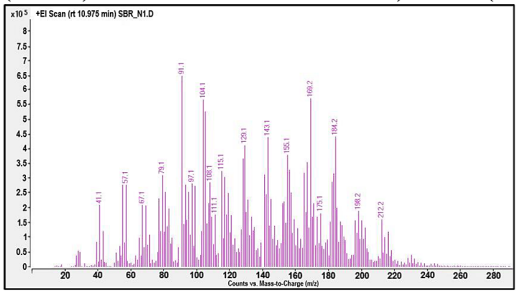
f) C12H18 (B trimer)



g) C12H18 (B trimer)



h) C12H14 (SB hybrid dimer)



i) C16H12 (SBB hybrid trimer)

Figure 3. Mass spectra of pyrolytic decomposition products of the reference material SBR – reference mass spectra [18]

control, specifying the values of the acceptance number Ac and rejection number Re.

As a result of chromatographic analyses, chromatograms were obtained for each of the analytical samples of SBR rubber granulate. Examples of total ion chromatograms (TIC) for two parallel analytical samples are presented in Figure 4.

By comparing the chromatograms for the analytical samples with the chromatogram of the reference material, characteristic peaks of SBR pyrolytic decomposition products were observed at corresponding retention times. The identified peaks for the tested samples corresponded to the peaks from the analysis of the SBR reference material.

Comparison of the mass spectra obtained for the tested rubber granulate with the reference spectra revealed the presence of characteristic SBR pyrolytic decomposition products, namely: 1,3-butadiene, toluene (TO), 4-vinylcyclohexene (D), styrene (S), α-methylstyrene, C12H18(B trimer), C12H14 (SB hybrid dimer), C12H14 (SB hybrid dimer), and C16H12 (SBB hybrid trimer) in all analytical samples. Based on these test results, it was concluded that the material of the samples taken from the tested batch of polymer materials was SBR granulate.

Analysing the test results in light of the adopted quality control criteria, it was found that the tested batch of 60 transport packages of rubber granulate from recycled car tires has a homogeneous SBR material.

MECHANICAL TESTS

Material for mechanical tests was prepared by pressing on a phM 63 hydraulic press. The pressing temperature was 160 °C, the pre-pressing time was 5 minutes. The process was carried out with a maximum pressure of 9.5 MPa for 5 minutes. The cooling time was 10 minutes. The prepared material was presented in Figure 5.

Figure 5. Pressed plate

Density

The density was determined for 5 samples cut from the obtained plates with a thickness of 5 mm and a diameter of 35 mm. A view of the sample is shown in Figure 6. The dimensions were measured with a Mitutoyo IP67 digital caliper. The mass was determined using an Ohaus Adventurer Pro analytical balance, OHAUS Europe GmbH, Greifensee, Switzerland), following ISO 845 [21].The average density was 1.07 g/cm^3 with standard deviation = 0.05 g/cm^3.

Water absorption

Water absorption (W) tests were carried out and calculated according to ISO 62 [22]. Properly prepared samples (Fig. 6) were dried to constant mass (ms) at a temperature of 30 ± 20 °C, then immersed in a water bath at a temperature of 25 ± 2 °C for 24 h and weighed (mw). Water absorption was determined by weight according to the formula:

$$W = \frac{m_w}{m_s} 100\ (\%) \qquad (1)$$

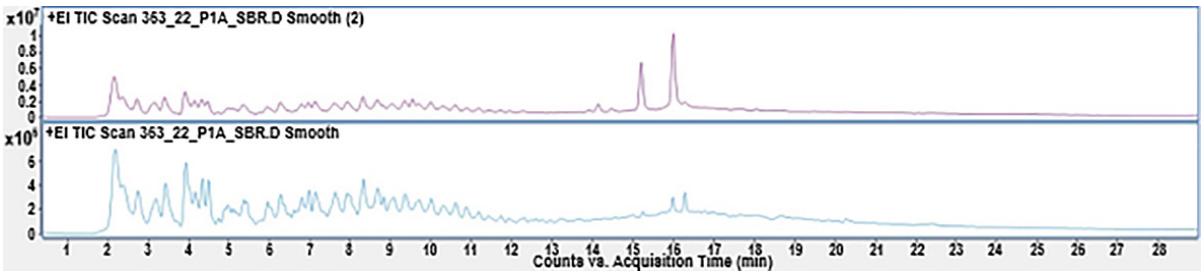


Figure 4. Examples of TIC chromatograms for samples of rubber granulate

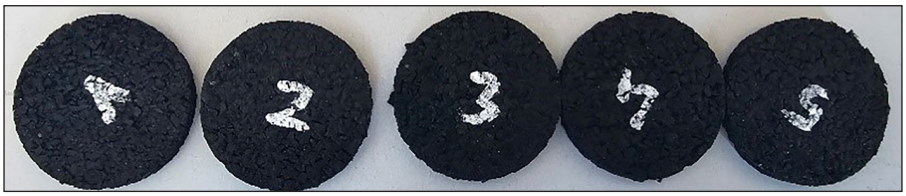


Figure 6. View of prepared samples

The average absorbency of the tested material was 1.68% with a standard deviation of 0.61%, which provides the appropriate features for materials used, among others, on playgrounds.

Static tensile strength

The tensile strength and strain at break were determined in the static tensile test. The tests were carried out in accordance with ISO 527-1 [23] on an AGX kN10D testing machine (Shimadzu Corporation, Kyoto, Japan) equipped with a DSES-1000 long-distance contact extensometer with a measuring gauge of 10 mm and cooperating with the Trapezium software. The tensile speed was 5 mm/min. The samples (Fig. 7) for testing were prepared by the cutting method. Figure 8 presents a stress-strain curve. The average strain at break was 55.51% (standard deviation = 3.22%) and the average tensile strength was 0.81 MPa (standard deviation = 0.06 MPa). The scatter of results, especially in the case of strain, was influenced by the sample structure, which caused gradual breaking. This is evidenced by the destruction of the samples – Figure 9.

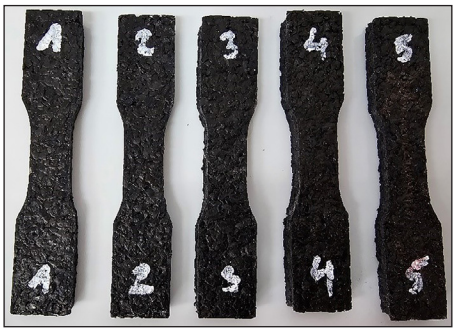


Figure 7. View of samples

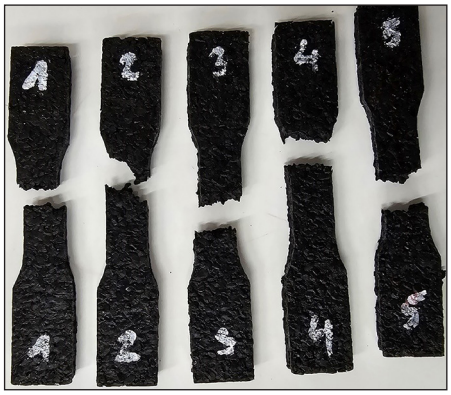


Figure 9. View of samples after tensile test

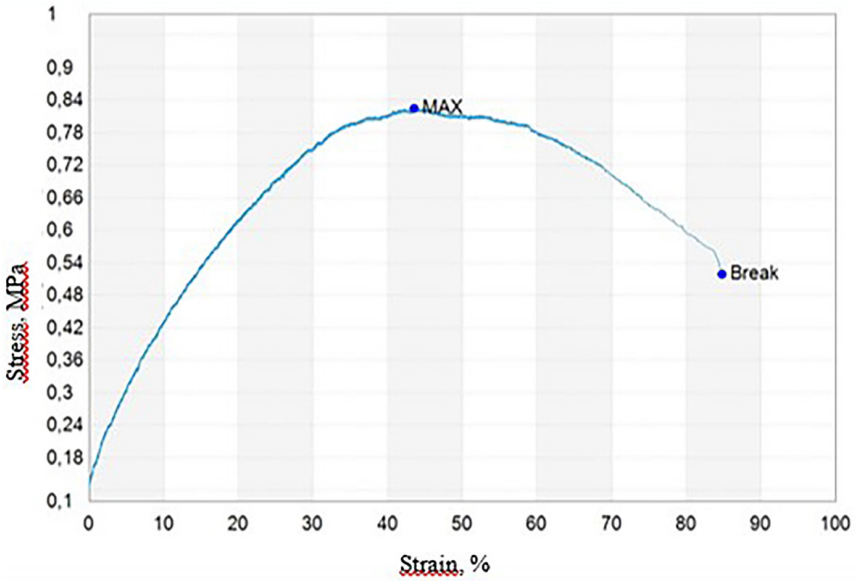


Figure 8. Stress-strain curve

Hardness

The hardness was determined by the Shore A method according to ISO 7619-1 using a Zorn Stendal (Stendal, Germany) durometer [24]. The tests were carried out on the samples shown in Fig. 1b, with 3 measurements taken on each sample. The result is an average of 15 measurements. The hardness was 53.4 °Sh A, and the standard deviation = 3.2 °Sh A, which is about 6% of the average. The observed scatter of results is related to the structure of the tested material.

REBOUND RESISTANCE

Elasticity was determined according to ISO 4662 using a Shoba apparatus (Heckert, Chemnitz, Germany) [25]. Samples with dimensions 50 × 50 × 5 mm was tested three times, each time hitting a different spot with the weight. The impact speed was 2 m/s. The mass of the indenter was 250 g. The average rebound resistance was 23.8% and the standard deviation was 1.9%.

The testing procedure is presented in the flowchart – Figure 10.

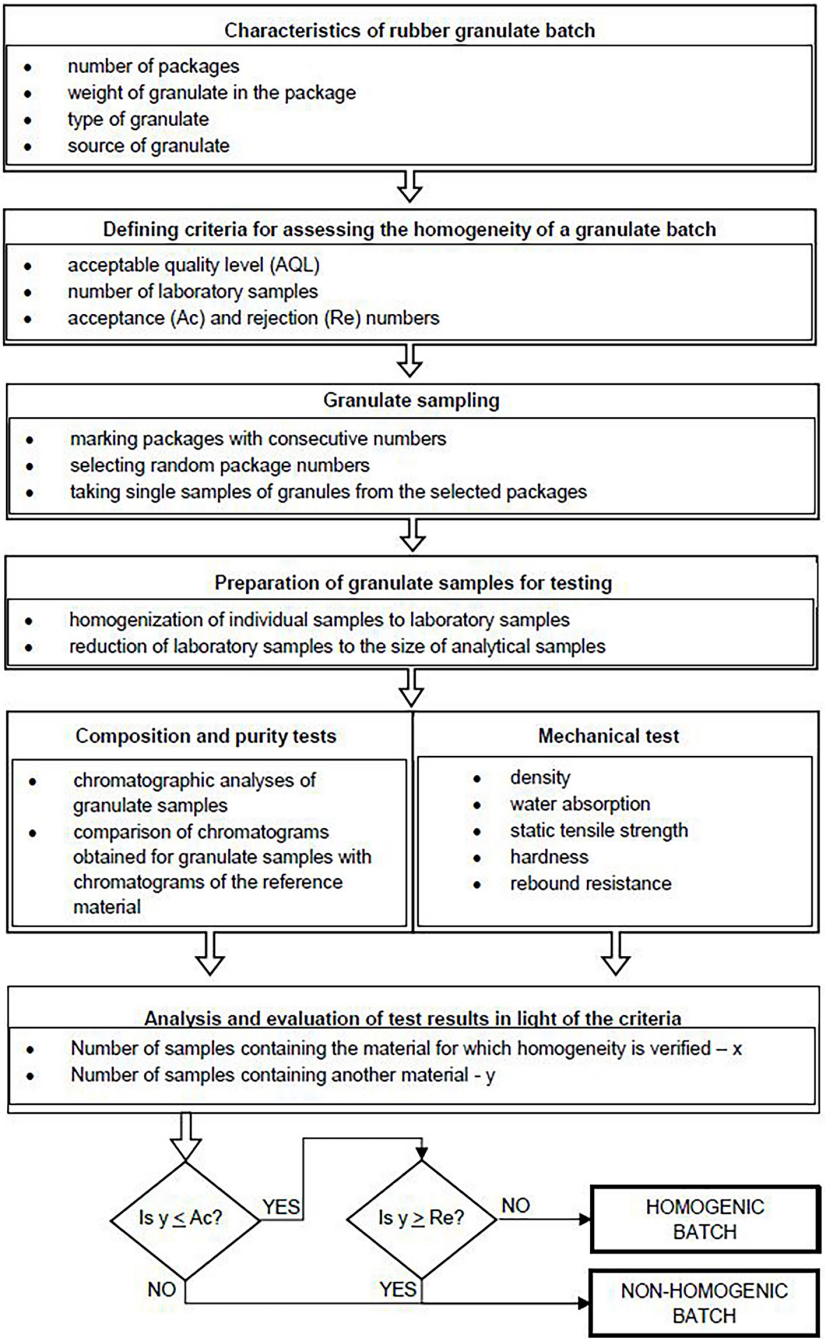


Figure 10. Flow chart of the testing procedure

CONCLUSIONS

Clear confirmation of the composition of polymer materials, especially those from recycling, is one of the fundamental conditions determining their suitability for further use. Identifying the types of materials making up a batch is a complex process that requires using the advanced analytical tools, such as the method of pyrolytic thermal desorption coupled with gas chromatography and mass spectrometry detection (Py/TD-GC-MS).

Due to the high mass of batches of polymer materials, the assessment of their homogeneity requires the use of statistical analysis tools, which enable the provision of the results regarding the material composition, forming the basis for taking decision regarding its acceptance or rejection. The presented test results confirmed the possibility of using the statistical acceptance sampling method (SKO) with an alternative assessment for this purpose. This type of control is based on a plan specifying, for a given batch size and control level, the required number of samples for analysis and, for the adopted AQL, the acceptance number (Ac) and rejection number (Re).

The method presented in the publication for assessing the homogeneity of the composition of rubber granulate batches can be applied to assess the homogeneity of large batches of other types of materials from recycling. The evaluation results, determining the percentage of undesired components in the batch, can serve as a basis for its acceptance or rejection if it fails to meet the requirements.

The presented method can also be applied to assess other parameters of the polymer materials batches, such as the content of substances harmful to the environment and human health. Using statistical analysis tools to determine the number of laboratory samples in this regard will help limit the scope of costly and time-consuming analyses of large batches of bulk materials.

The presented testing methodology of the determining material purity should be supplemented by tests of operational properties. The use of the type of materials for playground and gym covering requires determining the flexibility, water absorption, hardness, etc., which is related to the comfort of use. The study results of physicomechanical properties confirmed the suitability of the tested material for playground flooring, where low water absorption, hardness, and relatively high flexibility are required. The improvement of the indicated properties is possible by introducing additional modifications.

Despite its advantages, the developed method has limitations that require further analysis. These particularly concern the representativeness of the collected samples. In this case, even slight deviations in the sampling procedure can lead to significant differences in the results, which may introduce errors in the overall assessment of the material batch. Ensuring the representativeness of samples is especially challenging when analysing large batches of rubber granules, for instance, over 30 transport packages, each with a capacity of 1 ton, which can pose a control challenge in the construction of sports fields. To minimize this limitation, it may be necessary to improve the sampling method, including determining the sampling points at different levels of a big-bag package and selecting an appropriate sampler for this purpose.

Further development of the method should focus on incorporating the batch acceptance inspection with a numerical assessment based on the analysis of statistical measures determined for batch parameters, such as the content of undesirable substances in the selected samples relative to the criterion values established for the required quality level. When applying the method to assess purity of rubber granulate batches used in the construction of sports field surfaces, an important parameter is the content of toxic chemical substances, such as carcinogenic polycyclic aromatic hydrocarbons, which may be present in the materials from recycled car tires.

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