

The role of laboratory testing and sampling in ensuring material compliance and controlling the rubber production process

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Abstract. Laboratory testing plays a key role in ensuring the quality of the rubber production process. Quality has a direct impact on the efficiency and innovation of industrial enterprises. Control of the chemical structure of rubber at individual stages of production, carried out using advanced analytical methods such as FTIR (Fourier Transform Infrared Spectroscopy), DSC (Differential Scanning Calorimetry) and GPC (Gel Permeation Chromatography), enables the detection of any non-compliance. FTIR enables an early assessment of structural changes in the material, which is the foundation for further research. DSC is used to study the stability of polymers. Its main purpose is to analyse changes in the mechanical, physical and physicochemical properties of polymers with a controlled increase or decrease in temperature. The technological process of rubber production also depends on the molecular weight of the sample at a given stage of the process. The analysis was performed using the GPC method. By using the selected testing techniques, the rubber production process can be corrected promptly,

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minimising waste and improving the quality of the final product. Regular testing, including the identification of moisture content, eliminates problems that can prevent precise measurements and the ultimate functionality of the rubber. Based on the results of the tests, changes were made to the rubber production technology, introducing an additional drying stage which enabled not only the elimination of the observed problems but also the optimisation of the process and improvement of the quality of the final product. Such measures make it possible not only to enhance productivity but also to meet the growing demands of customers and remain competitive in a dynamic market.

Keywords: rubber, quality, FTIR, DSC and GPC

1. INTRODUCTION

The rubber industry is an important part of industrialised economies. It is impossible to imagine the development of sectors such as construction, automotive or household appliances production without the rubber sector. At the same time, rubber is a renewable raw material, which is particularly important for sustainable development (Boonmahitthisud and Boonkerd, 2021; Kohjiya, 2021).

In a rapidly growing rubber market, a key objective for any company is to achieve a competitive advantage through, e.g., improving its production processes (Setiawan, 2012). The literature (Myint Maung et al., 2023; Akhtar and Tsang, 2023; Lopes et al., 2017) on natural rubber production technology recognises the significant role of innovation as a driver of development in the rubber sector. Modern technical solutions make it possible to optimise vulcanisation processes and to improve the physical and chemical properties of rubber, which affects its durability and resistance to environmental factors (Sukkaneewat and Utara, 2022). Based on the research by Boon et al., (2022) the level of innovation in the rubber production area, similarly to other industries, also depends on the management strategy and the approach to investment in research and development (Asami et al., 2023). Despite the still dominant financially focused approach, more and more companies are recognising the benefits of cooperation with research centres. Laboratory tests play a key role in that context, with the task of not only monitoring rubber properties but also optimising the entire production cycle (Ansari, 2021).

Scientific literature on synthetic rubber production technology is dominated by studies focusing on general issues such as innovative synthesis methods, polymer structure modifications, and process parameter optimization. Particular emphasis is placed on increasing production efficiency, reducing operating costs, and limiting negative environmental impact (Fajczak-Kowalska, 2022). Against this background, there is a clear research gap concerning the rubber drying process, which, despite its fundamental importance for the quality and durability of the

material, remains marginally present in technical literature. Rubber drying is most often treated as a uniform, final stage of production, carried out after the material has been formed into sheets, blocks, or granules. In both natural and synthetic rubber production, it is not divided into preliminary and final phases, which limits the possibility of precisely controlling the parameters that affect the physicochemical properties of the product (Fajczak-Kowalska, 2022). This is also confirmed by the research of Tham et al. (2014), who describe the drying of rubber granules as an intensive thermal process requiring high energy inputs due to the low thermal conductivity of the material. Although various drying technologies are discussed, the process is treated as a uniform stage after forming, without separation into phases. Kumar and Singh (2023) focused mainly on optimizing drying parameters - such as temperature, humidity, and time - and on improving energy efficiency and the quality of the final product. However, there is a lack of analysis on the division of drying into phases, which limits the possibility of adapting the process to the specific characteristics of different types of rubber. A similar approach can be seen in the publication by Tan and Lim (2020), where drying appears as the final process after coagulation and filtration, without any indication of a step-by-step approach. The main focus is on the impact of drying on mechanical properties, not on its structural division. Obregon (2021) notes that laboratory studies on rubber drying are most often conducted retrospectively or at the final stage, which hinders their practical application in ongoing process control. Mathew et al. (2022) also point out in their review of rubber drying methods that the goal is to remove moisture after the material has been formed, and drying is described as a final technological process, without dividing it into phases. Aguele et al. (2021) compared different drying methods, showing differences in the quality of the rubbers obtained, but all were used as a single, final stage, without considering the effect of pre-drying on the structure of the material. Mathew et al. (2022) also treat drying as the final stage before packaging or vulcanization, focusing on the effect of temperature on mechanical properties, without analyzing the stages of the process.

Previous research on the production process of rubber - both natural and synthetic - has focused mainly on the stages of polymerization, mixing, vulcanization, and final processing. The rubber shredding stage, which often precedes further processing, has not yet been sufficiently analyzed in terms of the impact of moisture remaining in the material on the quality of laboratory analyses and the properties of the final product (Olejnik et al., 2019; Xiang, 2019; Tham et al., 2014). Despite the growing interest in effective rubber drying techniques, there is a lack of research in the world literature on the impact of moisture remaining after shredding styrene-butadiene rubber on the quality of spectroscopic and thermal analyses and on the processing properties of the material. The collected sources clearly confirm that in technical and scientific literature, the rubber drying process is treated as a uniform, final stage of production, carried out after the material has been formed. There is a lack of analyses dividing drying into preliminary and final

phases, which makes the presented approach - with an additional drying stage after shredding - innovative and potentially groundbreaking for the quality control and durability of rubber.

Thanks to modern testing methods, manufacturers are able to precisely analyse the physical and chemical properties of the material, such as elasticity, hardness or abrasion resistance. Advanced technologies such as FTIR spectroscopy or microscopic imaging make it possible to detect even the smallest defects accurately (Merriman et al., 2022; Craciun et al., 2020).

The introduction of an additional drying stage after shredding can significantly improve the technological process, improving both the quality of analytical data and the properties of the final rubber product. In the context of rubber production, FTIR and DSC techniques are of particular scientific importance—they enable quality control, monitoring of the vulcanization process, and structural analysis of the material. The automation of tests and the use of artificial intelligence in the analysis of results allow improvements to be implemented more quickly, resulting in increased production efficiency and reduced costs (Godoy, 2011).

The importance of regular quality audits cannot be ignored either, as they are an essential element in eliminating manufacturing defects. Regular audits allow the early detection of potential problems, the implementation of corrective actions and the maintenance of high standards in line with current industry norms. Audits make it possible not only to maintain quality at an appropriate level, but also to build lasting customer confidence in rubber-based products.

The paper takes a closer look at the issue of quality and its improvement in the rubber production process. With ever-increasing competition, quality in the business sector under discussion is treated as an important strategic objective and is considered a key management instrument. The purpose of this study is to demonstrate the importance of the use of testing techniques in the rubber production process and their impact on the quality of the final product and the efficiency of the process. The objective of the performed analyses was to identify key issues, such as moisture content preventing precise measurements, and to develop changes in the technology to eliminate them. The result was not only increased product reliability and functionality, but also optimisation of the entire production process. The article proves that regular tests (FTIR and DSC) support technological decision-making, leading to an increase in the competitiveness of the company and better adaptation to market expectations, inter alia.

2. MATERIALS, EQUIPMENT AND RESEARCH METHODOLOGY

A key element of quality assurance in the rubber production process is the analysis of compliance with the requirements of technical specifications, which is possible through the implementation of effective control and improvement measures. The aim of this work is to evaluate the effectiveness of control and measurement techniques in rubber production and their impact on product quality and process efficiency. Particular attention was paid to the drying

stage after rubber shredding and the use of FTIR and DSC methods in sample analysis. Early detection of material defects - thanks to proper sampling and sample preparation - allows for quick correction of technological parameters, which significantly contributes to the optimization of the entire production process. One of the most important aspects investigated as part of this study is the change in the chemical structure of the rubber during the production process. That parameter is checked three times at different technological stages, which allows the progress of the reaction to be controlled and ensures compliance with the established quality standards (Fig. 1). Of key importance in this respect is FTIR (Fourier Transform Infrared Spectroscopy), which provides information about the molecular structure of the tested samples by analysing the characteristic vibrations of chemical bonds (Caban, 2022; Ďurišová et al., 2024). Satisfactory results obtained in the FTIR analysis constitute the basis for performing further, more advanced analyses using DSC (Caban and Gnatowski, 2024) and GPC methods (Wei et al., 2022), allowing for the assessment of thermal properties and molecular weight distribution of the material, respectively.

The tests were performed using a sample of rubber produced by one of the leading Polish chemical companies. Styrene-butadiene rubbers are widely used in the automotive industry. They are used in the production of car tyres, inner tubes, conveyor belts, footwear, cables, hoses and other various technical rubber products.

Spectroscopic analyses were performed using the AVATAR 320 spectrophotometer with an ATR reflectance attachment and a diamond crystal. The spectra were recorded in the measurement range from 400 to 4000 cm^{-1} . A total of 64 scans per measurement were performed, with a resolution of 1 cm^{-1} . The preparation of samples for FTIR tests consisted of pressing the rubber sample to achieve a 2mm thick film, with a diameter of 1 cm. The pressing was carried out on a vulcanisation press, at a temperature of 100 °C and a load of 30 tonnes.

The thermal properties were analysed using the DSC method with the use of a Mettler Toledo scanning microcalorimeter, DSC – 1 type. DSC curves were recorded while the samples were heated at a rate of 10 °C/min. The mass of the samples ranged from 6 to 11 mg.

Gel permeation chromatography (GPC) was performed using the SHIMADZU gel chromatograph with a DawnHeleos II light scattering detector.

The production process of styrene-butadiene rubber is based on a well-established technological scheme, which includes the following stages: from monomer synthesis, through polymerization, coagulation, drying, and granulation, to mixing with additives, forming, vulcanization, and final quality control and packaging. This model, commonly used in industry, assumes a uniform drying process, carried out after polymer separation and before further processing stages. However, in light of the latest research and practical observations, there is a need to revise this approach. It is increasingly pointed out that treating drying as a one-off, final

stage may limit the possibilities of controlling the physicochemical properties of rubber, especially in terms of its durability, homogeneity, and susceptibility to further processing. An alternative technological scheme, which includes an additional drying stage after shredding the material and ongoing control of process parameters, is an innovative approach that can significantly affect the quality of the final product. By presenting a detailed technological diagram of the rubber production process (Figure 1), the sample-collection points crucial for the quality monitoring at various stages of production were marked. Indicating the points on the diagram enables a better visualisation of the quality control process for raw materials, intermediates and final products, enabling more accurate analysis and assessment of compliance with specification requirements.

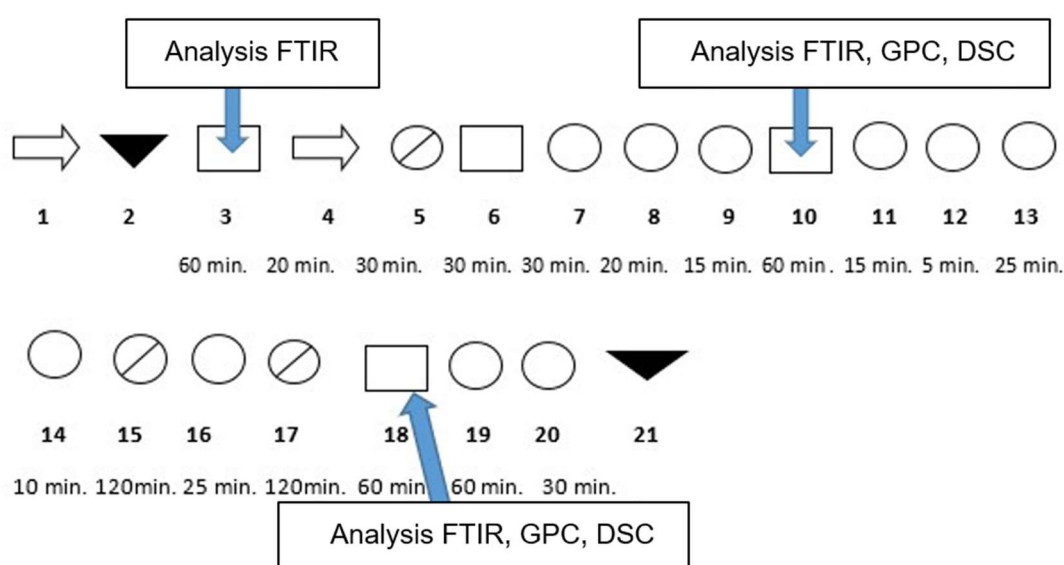


Figure 1. Diagram of the rubber manufacturing process in technological terms, including the analyses performed at the control stages. Key:

1. Delivery of raw material (latex) to the company.
2. Storage of the raw material (the storage time depends on customer needs and orders).
3. Initial quality control of the received raw material by the R&D department and assessment of compliance with technical conditions and relevant standards – 60 min.
4. Transport of the raw material to the production hall – 20 min.
5. Preparation of the process line installation to start rubber production – 30 min.
6. Entering appropriate parameters at control stations – 30 min.
7. Placing the raw material in the start-up tank and heating the content to the appropriate temperature – 30 min.
8. Preparation of a mixture containing latex, cross-linking agents, colouring agents and dosing the ingredients in appropriate quantities – 20 min.
9. Mixing the prepared mass – 15 min.
10. Sample collection for quality testing – 60 min.
11. Handling of the liquid compounds included in the mixture after receiving the results for the control parameters from the analytical laboratory – 15 min.

12. Weighing and dosing latex, cross-linking agents, antioxidants, flame retardants - 5min.
13. Chopping the resulting granulate – 25 min.
14. Washing the chopped rubber – 10 min.
15. Removal of water from the rubber by means of evaporation – 120 min.
16. Chopping the washed rubber – 25 min.
17. Drying the chopped elements – 120 min.
18. Sampling for quality testing and quality control – 60 min.
19. Forming ready-made rubber blocks – 60 min.
20. Transport of the finished product to the warehouse – 30 min.
21. Storage in the warehouse - the storage time depends on customer needs.

3. RESEARCH RESULTS

Infrared spectroscopy (FTIR-ATR) is the analysis of the structure of individual particles and the composition of mixtures. It is used to make sure that raw materials, intermediate compounds and final products meet the required specifications. Figure 2 shows the recorded FTIR-ATR spectra of the tested materials.

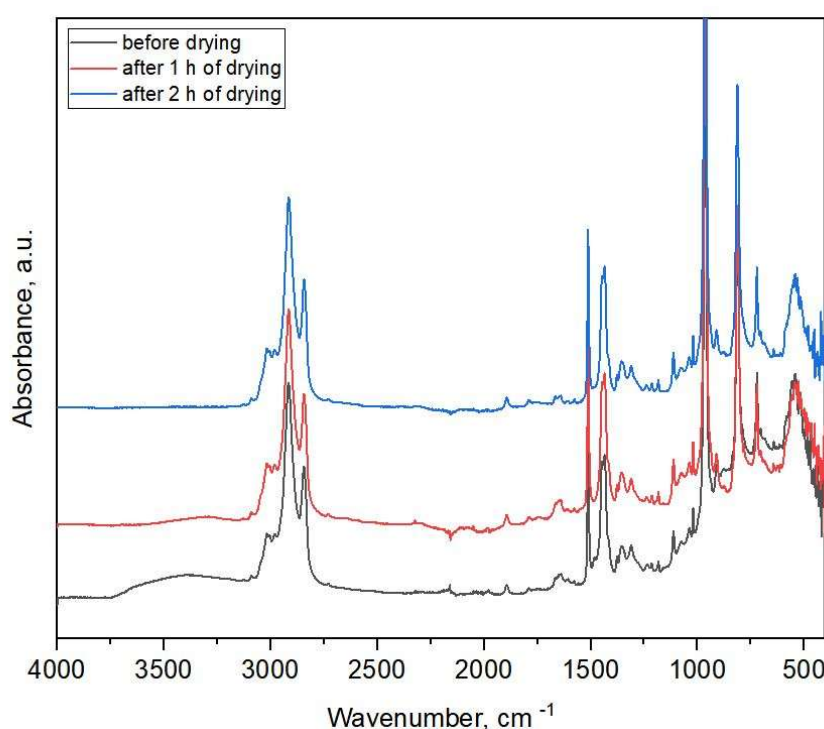


Figure 2. FTIR - ATR spectra of the tested rubber samples.

The rubber samples absorbed too much moisture during the synthesis. This is noticeable as an increase and broadening of the signal located around wave number 3500 cm^{-1} . Such a band corresponds to the swelling effect of the samples under the influence of moisture (Sarma et al., 2018). This is an undesirable effect. That phenomenon is typical of the initial stage of rubber

synthesis, when the solvent has not yet evaporated. To improve the quality of the FTIR-ATR spectrum, the sample preparation method was further developed and one more stage was added prior to the analysis. To eliminate moisture, before pressing and measuring the FTIR-ATR spectrum, the sample was dried at a temperature of 100 °C for 1 h. While analysing the spectrum of the sample after 1 h of drying, it could be seen that the moisture content decreased, but it was still clearly visible on the spectrum in the 3500 cm⁻¹ band. To eliminate the moisture completely, it was decided to extend the sample drying time to 2 h. After the FTIR-ATR measurement, the positive effect of increasing the sample drying time could be noticed. The moisture was completely eliminated from the sample, and there was no visible peak around wave number 3500 cm⁻¹ on the FTIR-ATR spectrum.

Figure 3 shows images illustrating the physical appearance of the rubber at a given stage with a different moisture content. The dark brown colour of the sample means that the sample contains a very large amount of moisture. As the drying time increases, the colour of the rubber changes (Figure 3c).

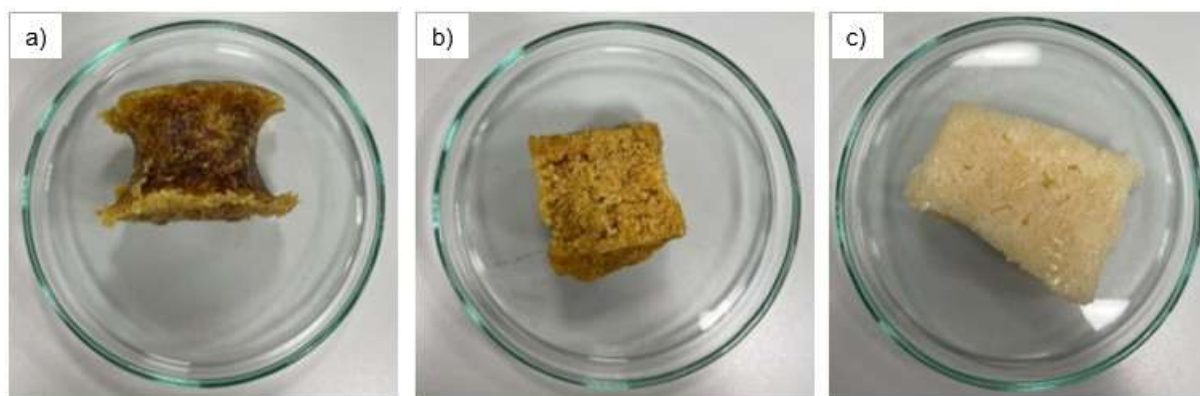


Figure 3. Rubber sample: a) before drying, b) after drying for 1 h, c) after drying for 2 h.

Another analysis that can be used to quickly control the rubber synthesis process is differential scanning calorimetry (DSC) (Ribba et al., 2019). This technique is used to test the stability of polymers. The DSC technique makes it possible to analyse changes in the mechanical, physical and physicochemical properties of polymers with a controlled increase (heating) or decrease in temperature (cooling). During heating of the sample, a number of physical and chemical changes occur, including cross-linking, solvent release, changes related to the glass transition temperature, melting and decomposition. The temperature at which the above-mentioned processes occur provides a lot of valuable information about the chemical structure of the tested material. The DSC method is a very fast and precise measurement technique, and only a small

amount of material is required to perform it. The example is rubber thermograms recorded at different stages of rubber synthesis (Figure 4).

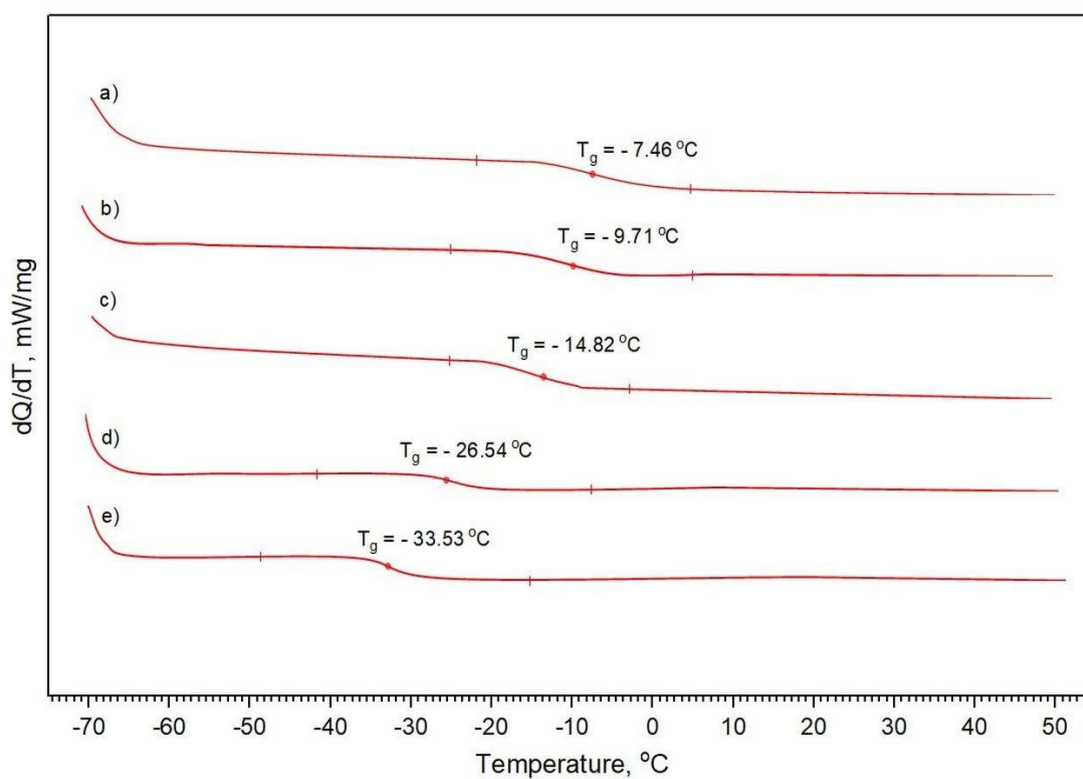


Figure 4. Thermograms recorded during the rubber synthesis process: a) after the initiation of the installation process, b) at the second production stage, c) at the third production stage, d) at the fourth production stage, e) at the final production stage.

The glass transition temperature (T_g) is an important physical property of rubber materials, as it determines the temperature at which the material transitions from an elastic phase, characterised by a rubbery consistency, to a rigid and brittle phase. This phenomenon is crucial for determining the mechanical properties and thermal stability of rubbers under various operating conditions (Liao et al., 2024). By analysing the DSC thermograms recorded during the rubber synthesis process, it can be seen that at the initial production stage, the rubber is characterised by a rather high glass transition temperature, approximately -7.46 °C. The glass transition temperature of the rubber decreases with the duration of synthesis. This is due to the incorporation of the main component into the raw material particles, complete evaporation of the solvent and polymer cross-linking. The desired result is a glass transition temperature of approximately -30 °C (Figure 4e). For many industrial applications, rubbers with a glass transition temperature of around -30 °C provide good flexibility at moderately low temperatures

while maintaining adequate mechanical strength (Changfei et al., 2019; Johnston and Shen, 1969).

The technological process of rubber synthesis also depends on the molecular weight of the sample at a given stage of the process. The analysis was performed using gel permeation chromatography (GPC) (Tianming et al., 2015). This is a type of exclusion chromatography, which is an analytical technique that allows the determination of the type of molecules based on their size. The process consists of the separation and differentiation of molecules with different hydrodynamic diameters in a porous column bed at a specific retention time. Particles smaller than the diameter of these pores are able to penetrate into the grains, while larger ones are completely excluded and, during leaching, move between the grains of the bed towards the column outlet. Since all particles are subject to diffusion, the smaller ones that had entered the pores escape to the outside after some time and are transported by the mobile phase towards the column outlet. The residence time of the particles inside the pores is proportional to their size. The smaller the particles, the longer they are retained inside the pores and later washed out by the eluent. Figure 5 presents graphs showing the molecular weight distribution expressed in g/mol for rubber samples from the beginning of synthesis to its completion.

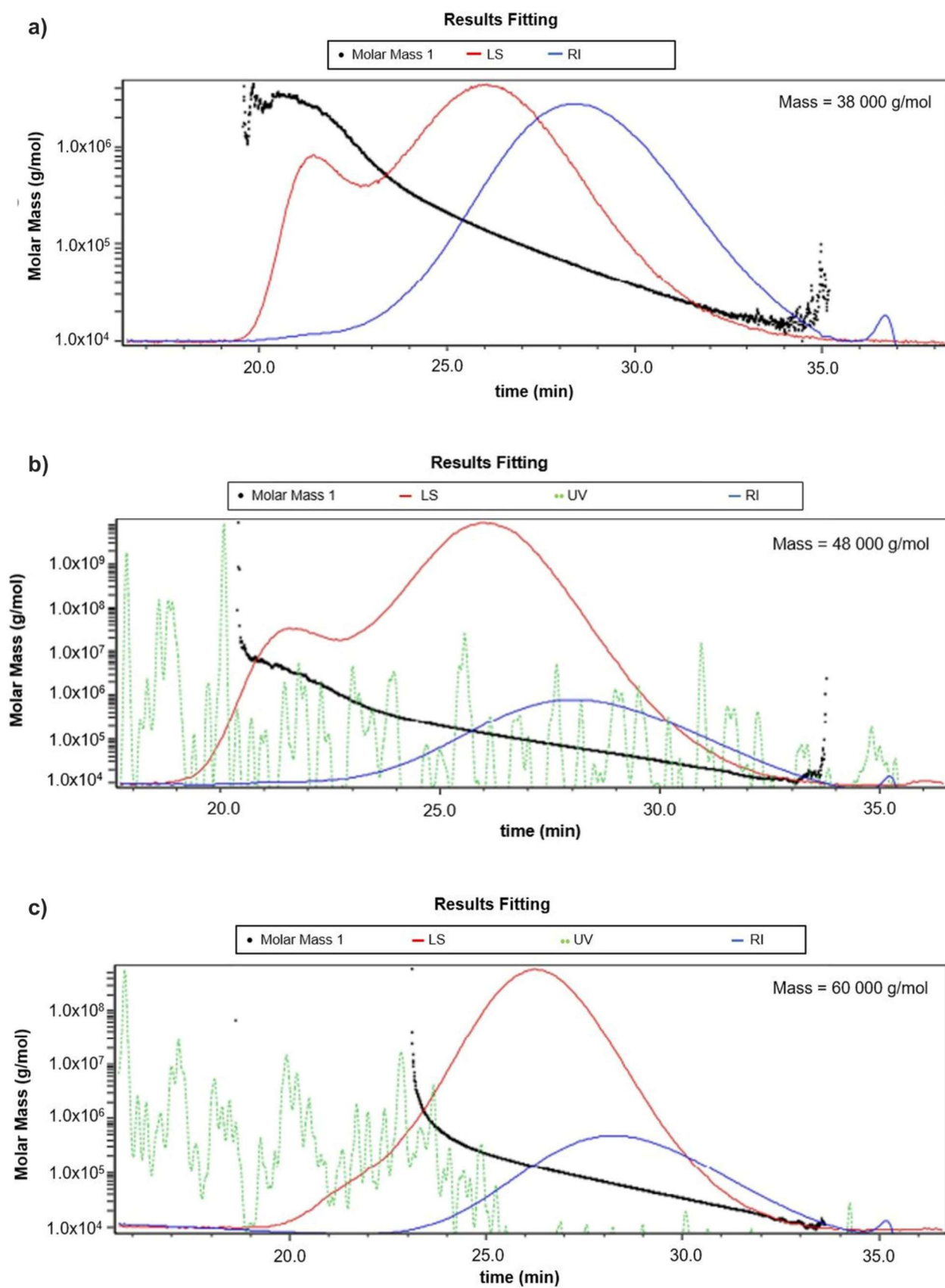


Figure 5. Molecular weight distribution at different stages of rubber synthesis: a) 1st synthesis stage, b) 3rd synthesis stage, c) 5th synthesis stage.

Initially, the peak is bimodal, indicating that there are two free compounds in the sample. This means that not all other components were incorporated into the styrene structure at the initial stage. By observing the molecular weight distribution at individual stages, it can be seen that the smaller peak is absorbed into the larger one, creating a unimodal peak. The expected mass to achieve process stability and complete synthesis is approximately 60 000 g/mol. The molecular weight of styrene-butadiene rubber is not a constant value and depends on the method of its production and specific application. It can range from several thousand (Kim et al., 2021) to several hundred thousand (Mills and Giurco 1976). To obtain that, different types of substances are added to the synthesis mixture, including: initiators, catalysts. This is of great importance as during the process, the rubber must not polymerise, a liquid state must be maintained. For the purpose of the analyses, a sample is collected from the reactor and subjected to drying and rolling to remove moisture. For comparison, the appearance of samples at the first and last stages of the technological process is presented in Figure 6.

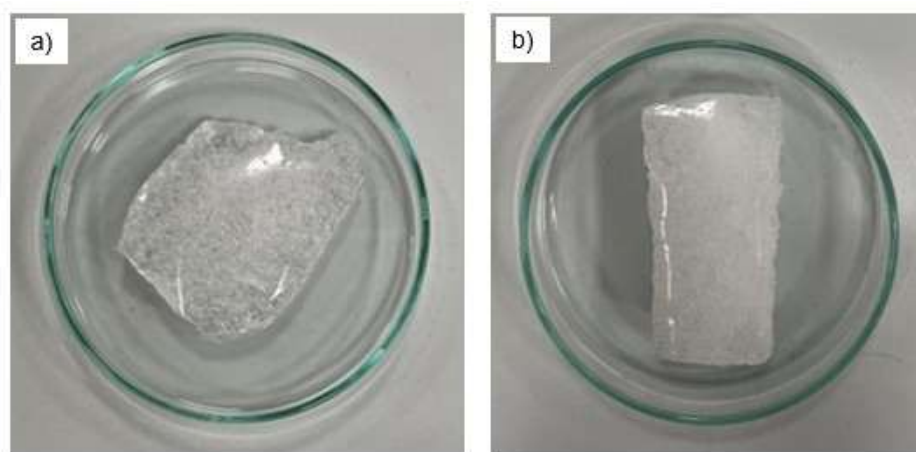


Figure 6. Rubber samples collected at the first and last stages of synthesis.

The sample collected at the first stage of rubber synthesis (Figure 6a) shows high transparency and air bubble content, which indicates the moisture content and loose structure of the chemical compound. In contrast, the sample collected at the last stage of synthesis (Figure 6b) is more bound, its colour is clearer. This is due to the polymerisation of the rubber with the other components of the mixture used for synthesis. Such rubber is harder, more physically and mechanically durable.

When analysing a series of samples collected during the technological process, certain correlations are observed, specific to the rubber type. In addition to the moisture content due to the presence of solvent, the qualitative composition of the rubber is also of great importance. Table 1 summarises the data for the rubber samples relative to the content of polybutadiene and the glass transition temperature.

Table 1. Summary data for rubber samples relative to the polybutadiene content and glass transition temperature.

Sample name	polybutadiene %	T_g [°C]
Rubber 1	9.5	-94.92
Rubber 2	10.06	-94.59
Rubber 3	10.51	-94.47
Rubber 4	11.25	-94.36
Rubber 5	12.4	-93.67

The samples are repeatedly subjected to FTIR (Figure 7) and DSC (Figure 8) analyses throughout the process.

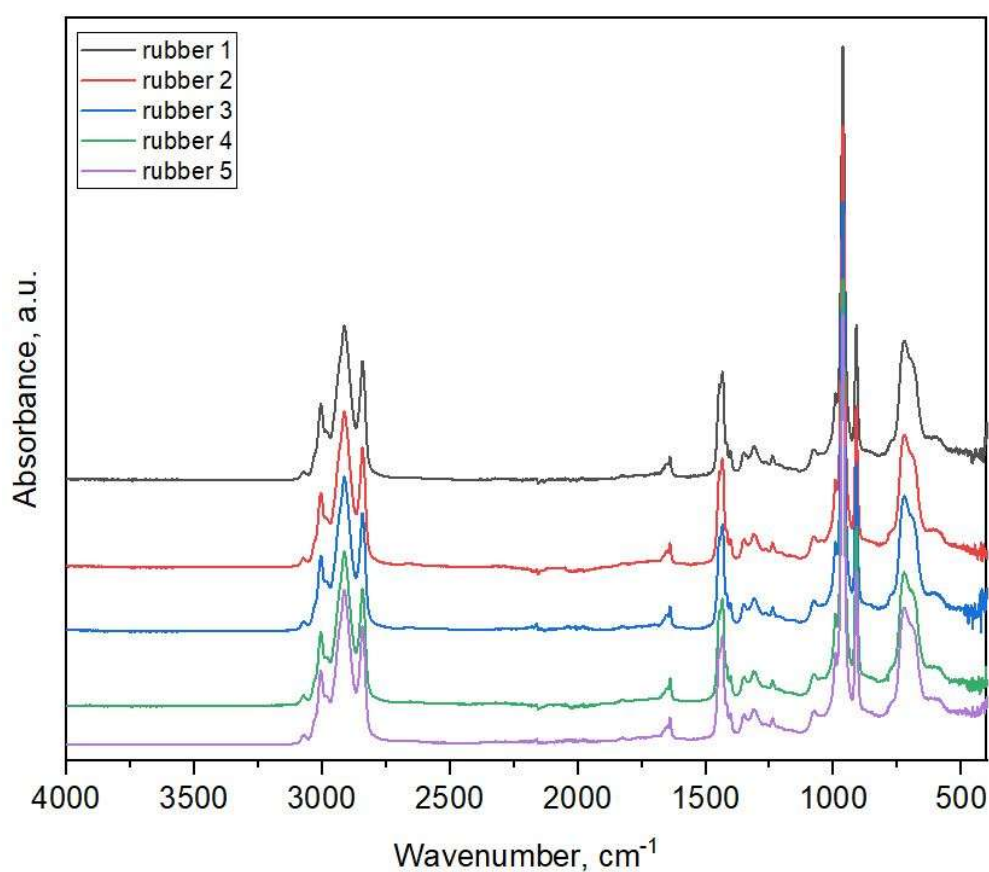


Figure 7. FTIR - ATR spectra for 1 – 5 rubber samples (with different polybutadiene content, %).

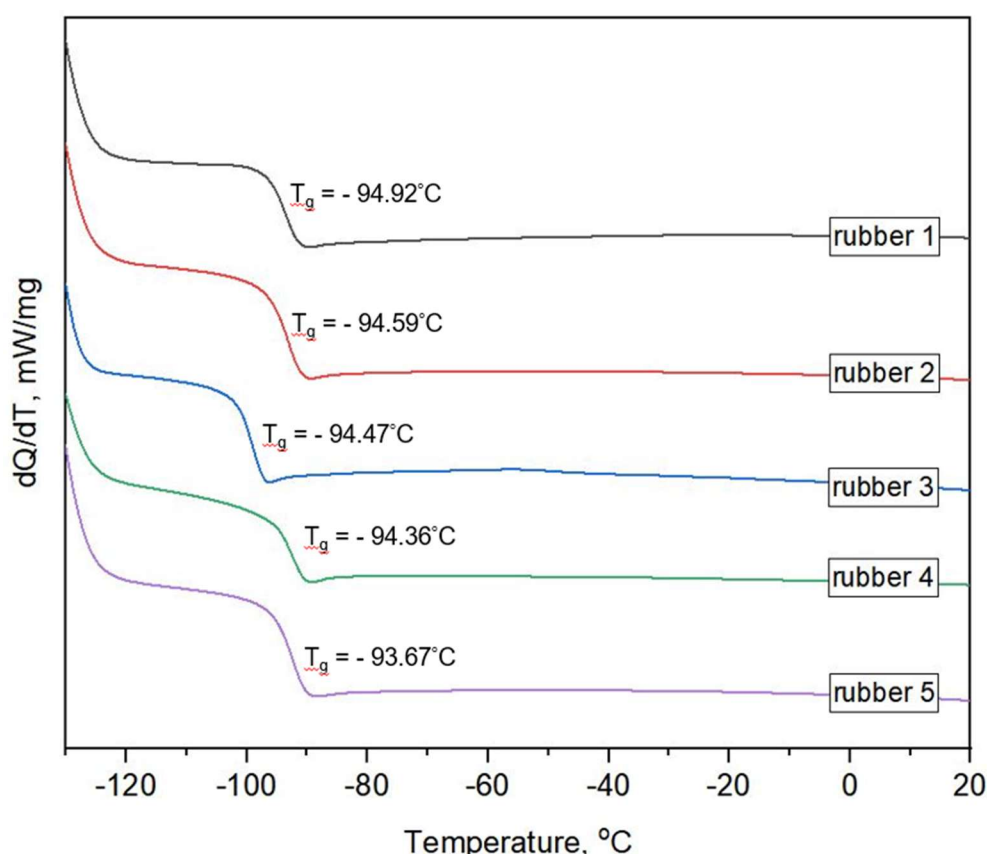


Figure 8. DSC thermogram for 1 – 5 rubber samples (with different polybutadiene content, %).

Based on the FTIR-ATR and DSC results, it can be determined whether the rubber synthesis process is running correctly. The higher the polybutadiene content in the sample, the higher the glass transition temperature. For polybutadiene rubbers, these are the control parameters. During the process, the aim is to stabilise the polybutadiene, where the maximum is 20%. The glass transition temperature must not be higher than -90 °C. This is due to the chemical structure of polybutadiene rubbers and the physical properties of their polymer chains. Polybutadiene does not contain rigid chemical groups, and its chains are characterised by a high degree of rotational freedom. As a result, the material exhibits high elasticity and high mobility of molecular segments (Gündüz et al., 2021). In the case of styrene rubbers, the control parameter is the share of styrene in the rubber structure. It indicates the degree of styrene incorporation into the chemical structure of the compound, which is very important for the further course of the process. When comparing the FTIR analysis with DSC, a correlation is observed that with decreasing styrene content, the glass transition temperature decreases (Figure 9).

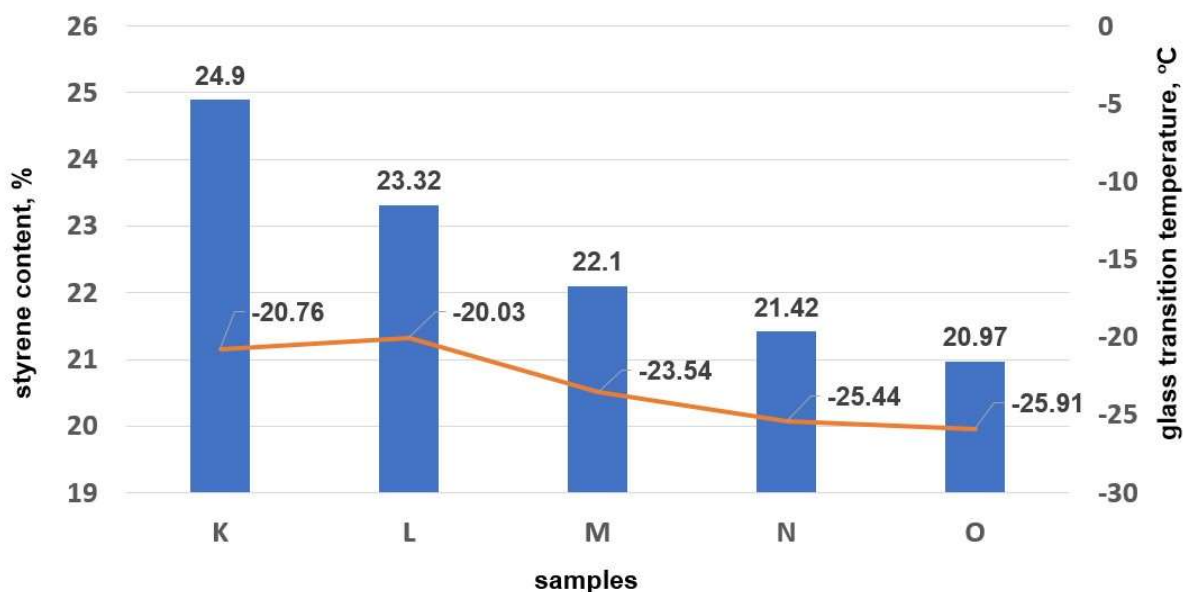


Figure 9. Correlation of the percentage of styrene content with the glass transition temperature.

The chemical structure of styrene monomer contains a rigid aromatic (phenyl) ring, which significantly limits the freedom of segmental movement of the polymer chain. The presence of these rigid structural units increases hardness and abrasion resistance. Reducing the proportion of styrene in the composition results in mixtures with reduced rigidity, which are also characterised by reduced mechanical resistance (Wang et al., 2021).

CONCLUSIONS

Laboratory tests during the technological process are an integral part of every product synthesis. This has a huge impact on the process improvement and the final quality of the product. Precise determination of the sample-collection points and the analysis sites ensures effective control of manufacturing processes and facilitates identification of potential problems that may affect the quality of the final product. Sampling at individual stages and the use of fast measurement techniques such as FTIR or DSC allow a rapid response to changes in parameters or the addition of a required component to the synthesis mixture. FTIR-ATR analysis revealed the presence of excessive moisture in the rubber samples, visible as an intense absorption band in the $\sim 3500\text{ cm}^{-1}$ range, corresponding to -OH groups. In order to improve the quality of the spectrum and increase the precision of the measurement, an additional stage of drying the samples before compression was implemented. Drying at $100\text{ }^{\circ}\text{C}$ for 1 hour reduced the moisture content, but the signal was still present. In the case studied, extending the drying time to 2 hours resulted in the complete disappearance of the water band, confirming the

effectiveness of the modification. This action significantly improved the quality of the spectroscopic data and enabled more precise monitoring of the technological process parameters. Such an approach allows more efficient improvement of the production process, minimises the risk of errors and material loss, as well as increases flexibility in responding to changing market requirements.

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