

OPTIMIZATION OF PROCESS OF SIEVE ANALYSIS OF RUBBER WASTE FOR PREPARATION OF ACTIVE RUBBER PARTICLES

BSc. Mohammad Shaqib Saifi

Master's Thesis
2024



Tomas Bata University in Zlín
Faculty of Technology

Tomas Bata University in Zlín
Faculty of Technology
Department of Polymer Engineering

Academic year: 2023/2024

ASSIGNMENT OF DIPLOMA THESIS

(project, art work, art performance)

Name and surname:	Mohammad Shaqib Saifi
Personal number:	T210064
Study programme:	N0722A130002 Polymer Engineering
Type of Study:	Full-time
Work topic:	Optimization of Process of Sieve Analysis of Rubber Waste for Preparation of Active Rubber Particles

Theses guidelines

Theoretical part

Methods of recycling rubber products
Methods of separation of ARP
Usage of ARP in industry

Practical part

Sieve analysis of the rubber waste
Analysis of individual fractions
Results and Discussion, Conclusions

Form processing of diploma thesis: **printed/electronic**
Language of elaboration: **English**

Recommended resources:

Asaro, L. et al. Recycling of rubber wastes by devulcanization. *Resources, Conservation & Recycling*, 2018, 133, 250-262
Grigoryeva, O. et al. Ground tire rubber (GTR) reclamation: Virgin rubber / reclaimed GTR (re)vulcanizates. *Rubber Chemistry and Technology*, 2004 DOI: 10.5254/1
Hoyer, S. et al. Technology comparison for the production of fine rubber powder from end of life tyres. *Procedia Manufacturing*, 2020, 43, 193-200
& another licensed resources (book, journals, etc.)

Supervisors of diploma thesis: **Ing. Martin Stěnička, Ph.D.**
Centre of Polymer Systems

Date of assignment of diploma thesis: **January 2, 2024**
Submission deadline of diploma thesis: **May 10, 2024**

prof. Ing. Roman Čermák, Ph.D. m.p.
Dean

L.S.

Ing. Jana Navrátilová, Ph.D. m.p.
Head of Department

In Zlín March 4, 2024

MASTER'S THESIS

AUTHOR'S DECLARATION

I take cognizance of the fact that:

- my Master's thesis will be stored in electronic form in the university information system and will be available for viewing;
- my Master's thesis fully adheres to the Act No. 121/2000 Coll. on Copyright and Related Rights and on Amendments to Certain Acts (Copyright Act), as amended, in particular to § 35 Paragraph 3;
- in accordance with § 60 Paragraph 1 of the Copyright Act, Tomas Bata University in Zlín is entitled to conclude a licence agreement on the utilisation of a school work within the scope of § 12 Paragraph 4 of the Copyright Act;
- in accordance with § 60 Paragraph 2 and 3 of the Copyright Act, I may use my work – Master's thesis – or grant the licence for the utilisation thereof to another party only with prior written consent by Tomas Bata University in Zlín, which is in such a case entitled to claim from me an appropriate contribution to the reimbursement of the costs incurred by Tomas Bata University in Zlín due to the creation of the work (up to the full amount of this cost);
- if a software was provided for the preparation of the Master's thesis by Tomas Bata University in Zlín or by other entities only for study and research purposes (i.e. for non-commercial use), the results of the Master's thesis cannot be used for commercial purposes;
- if the output of the Master's thesis is a software product, the source codes and/or the files of which the project is comprised are considered as an inseparable part of the thesis. Failure to submit this part may be a reason for failure to defend the thesis.

I declare

- that the Master's thesis has been solely the result of my own work and that I have cited all the sources I had used. In case of publication of the results, I will be listed as a co-author.
- that the submitted version of the Master's thesis and the version uploaded in electronic form in the IS/STAG system are identical in terms of their content.

In Zlín, on 10th May 2024

Name and surname of student: Mohammad Shaqib Saifi

.....

Signature of student

What is not started today is never finished tomorrow.

-Johann Wolfgang von Goethe

ABSTRAKT

Jednou z možností, jak využít pryžové výrobky, zejména pneumatiky, na konci jejich životnosti, jsou aktivní pryžové částice. Faktory, které ovlivňují výsledné vlastnosti pryžových částic pro další použití, jsou zejména jejich rozměry. Pryžové částice vyrobené jako druhotné suroviny z použitých pryžových výrobků mohou najít nové uplatnění v širokém spektru aplikací, pokud dosahují požadované kvality. Optimalizovaný postup výroby pryžových částic zahrnuje volbu vhodných zdrojů pryžového odpadu, optimalizaci podmínek zpracování a dosažení požadovaných vlastností. Výsledky této studie mohou přispět k efektivnější recyklaci a opětovnému použití pryžového odpadu, což by mělo mít pozitivní dopady na životní prostředí a ekonomiku.

ABSTRACT

Active rubber particles are one of the ways to use rubber products, especially tires, at the end of their useful life. Factors that influence the resulting properties of rubber particles for further use are mainly their dimensions. Rubber particles produced as secondary raw materials from used rubber products can be re-used in a wide range of applications if they reach the required quality. The optimized procedure for the production of rubber particles includes the selection of suitable sources of rubber waste, the optimization of processing conditions and the achievement of the desired properties. The results of this study can contribute to more efficient recycling and reuse of rubber waste, which should have positive effects on the environment and the economy.

ACKNOWLEDGEMENT

Here, it's my pleasure to thank everyone who helped with the completion of this thesis, I would like to extend my deepest appreciation.

I want to start by expressing my gratitude to my supervisor, Martin Stěnička, who has been a fantastic source of advice and support throughout the thesis. He has provided me with priceless advice, recommendations, and comments, and I appreciate them sharing his knowledge with me.

My sincere thanks go to Sanjoy Datta, who supported me throughout the thesis and assisted me with my experimental work. His support has been invaluable in helping me improve my work.

Without a doubt, I want to close by thanking my friends for their unwavering support.

I hereby declare that the print version of my master's thesis and the electronic version of my thesis deposited in the IS/STAG system are identical.

CONTENT

MASTER’S THESIS	4
AUTHOR’S DECLARATION	4
INTRODUCTION	10
I. THEORETICAL BACKGROUND	15
1. WHAT IS NATURAL RUBBER	16
2. USES OF RECYCLED RUBBER	18
2.1. TYPES OF RUBBER	18
2.2. UTILIZATION OF RUBBER IN TYRE MANUFACTURING	19
3. RECYCLING OF RUBBER (TYRES)	21
3.1. IMPORTANCE OF RECYCLING OF RUBBER (TYRES)	22
3.2. ADVANTAGES OF RECYCLING RUBBER	22
3.3. DISADVANTAGES OF RECYCLED RUBBER	23
3.4. ALTERNATIVE USES FOR RECYCLED RUBBER	23
4. METHODOLOGY OF RECYCLING	24
4.1. GRINDING TECHNOLOGIES FOR WASTE TYRE RUBBERS	24
4.2. CHALLENGES AND FUTURE TRENDS RELATED TO GROUND TYRE RUBBER	26
5. TECHNIQUES FOR RECYCLING THE RUBBER	27
5.1. MECHANICAL GRINDING	27
5.2. VULCANIZATION AND DEVULCANIZATION	28
5.3. RECLAIMED RUBBER	29
5.4. SIEVING PROCESS	29
II. EXPERIMENTAL PART	31
6. PURPOSE OF THE WORK	32
7. MATERIAL	33
8. METHODS	34
8.1. SIEVING PROCESS	34
8.1.1. Optimization of time of sieving	34

8.1.2. Optimization of the weight of the initial sample for sieving	34
8.1.3. Sample collection for modification.....	35
8.2. DRYING PROCESS	36
8.3. ACETONE TREATMENT	38
9. CHARACTERIZATION OF RUBBER PARTICLES.....	41
9.1. OPTICAL MICROSCOPY	41
9.2. THERMOGRAVIMETRIC ANALYSIS	42
9.3. INFRARED SPECTROSCOPY	42
10. RESULTS AND DISCUSSION.....	45
10.1. OPTIMIZATION FOR THE TIME OF SIEVING	45
10.2. OPTIMIZATION THE MASS OF THE RUBBER POWDER TO BE TAKEN FOR SIEVING	47
10.3. SIEVING OF THE ORIGINAL PARTICLES UNDER THE OPTIMIZED CONDITIONS	49
10.4. RE-SIEVING OF THE COLLECTED PARTICLES UNDER THE OPTIMIZED CONDITIONS	50
10.5. DRYING/RE-SIEVING OF THE COLLECTED PARTICLES UNDER THE OPTIMIZED CONDITIONS	51
10.6. ACETONE TREATMENT/RE-SIEVING OF THE COLLECTED PARTICLES UNDER THE OPTIMIZED CONDITIONS	52
10.7. DRYING/RE-SIEVING OF THE COLLECTED PARTICLES UNDER THE OPTIMIZED CONDITIONS	53
10.8. OPTICAL MICROSCOPY	54
10.9. TG ANALYSIS	57
10.10. INFRARED SPECTROSCOPY	59
CONCLUSIONS.....	62
LIST OF FIGURES	69
LIST OF TABLES	71
LIST OF SYMBOLS	72

INTRODUCTION

Rubber materials, originating from natural and synthetic sources, are important components in many industrial applications due to their distinctive features, such as elasticity, durability, and resistance to abrasion and corrosion. However, when the products become old and no longer useful for the contemplated end uses, then they need to be discarded and replaced by new products. These discarded rubber products may be recycled in various ways. One of the suitable outlets is to first grind the rubber wastes by suitable grinding techniques and then use a part by weight of the ground rubbers as fillers in pristine rubbers. This adds to the environmental benefits and helps to maintain the recycling process. The particle size and their distribution in each mass of ground rubber to be used for recycling have a significant impact on the performance of recycled rubber-based goods. Examples of ground rubber particles are shown in Fig. 1. Since there are size and weight distribution in each mass of obtained ground rubber it becomes necessary to go for quality assurance and control throughout the manufacturing process.



Fig. 1 Different particle size waste rubber [1]

The generated rubber wastes usually are from discarded tyres called end-of-life (EOL) tyres. The rubber contents of these tyres are mostly a blend of natural rubber, styrene-butadiene rubber and cis-polybutadiene rubber. Amongst these three rubbers, natural rubber alone contributes to a significant mass per cent of the total mass of the rubbers.

Natural rubber is generally recognized for its non-biodegradability, which raises worries about its possible influence on the environment. Rubber waste management creates substantial

environmental issues, and novel recycling and reuse technologies are needed. Different types of recycling methods are shown in Fig. 2 life cycle of tyres. One such process involves the production of rubber particles, which are used in a variety of applications such as fillers for pavements, sports grounds, and road construction and building materials.

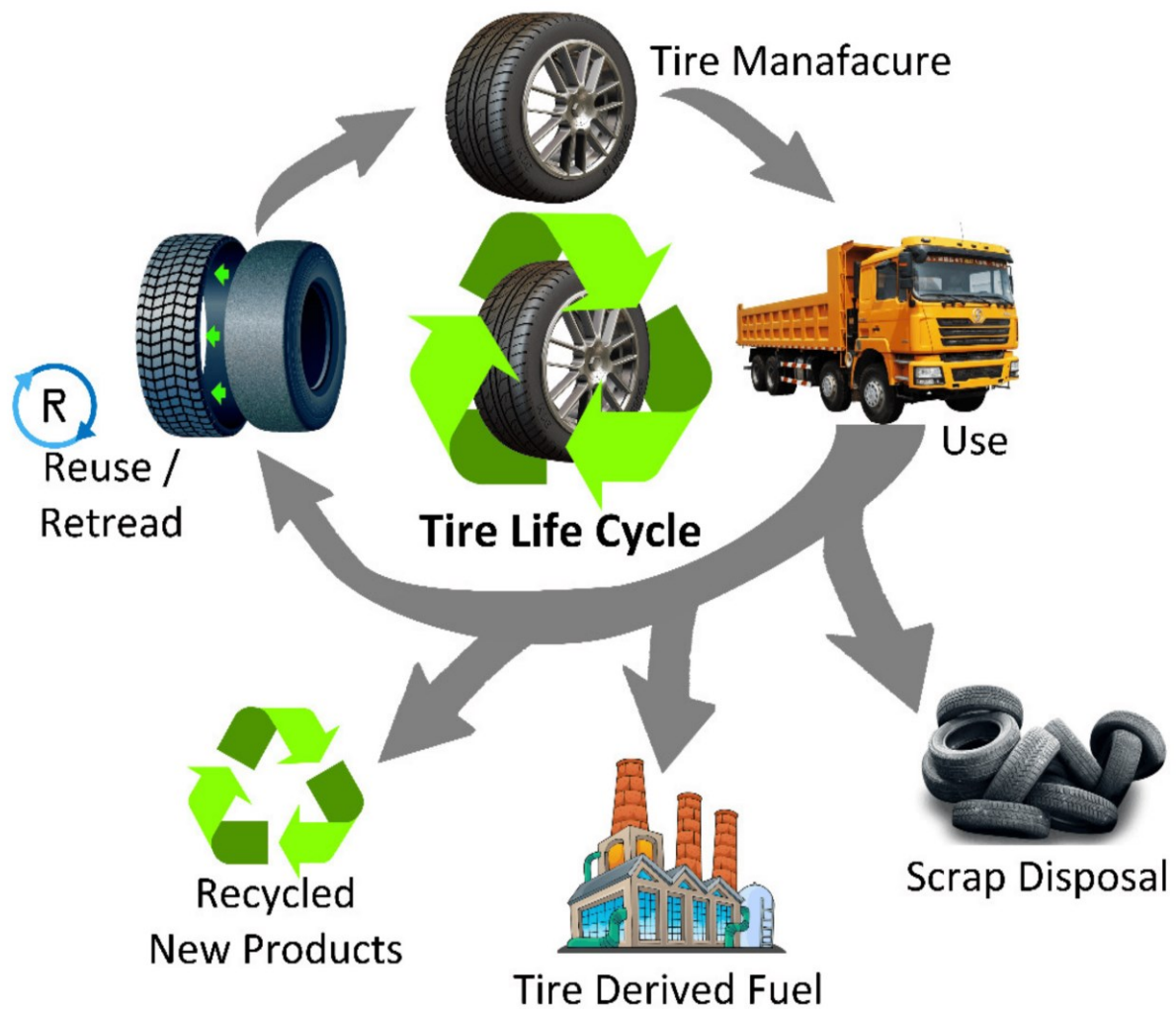


Fig. 2 Waste tyre recycling production [2]

Tyres, footwear, and industrial equipment are just a few examples of goods that employ rubber since it is a very flexible material. Rubber recycling can assist in lessening the impact rubber waste has on the environment, which is a major problem and can be identified in Fig. 3 showing the increase in the utilization of rubber over the past three decades worldwide.

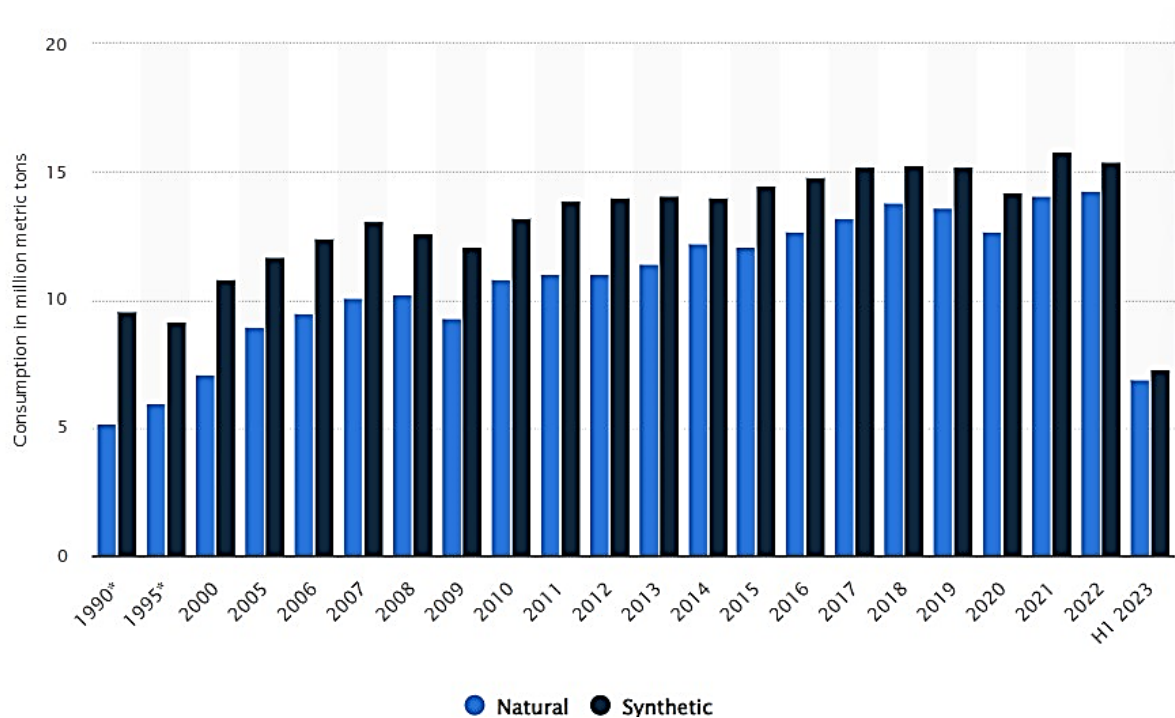


Fig. 3 Consumption of different types of rubbers in metric tons (= 1,000 kg) [3]

Rubber recycling may lessen waste and preserve resources, but there are also certain difficulties and issues with the procedure [4]. Rubber recycling has several significant drawbacks, such as:

Contamination: Rubber goods frequently contain different additives, such as fillers, colours, and adhesives, which can make it challenging to separate and recycle the rubber. The quality of recycled rubber can also be impacted by contamination from other materials like metal, cloth, or plastic. As shown in Fig. 4 the dumping of tyre waste is very expensive for recycling due to the mixed type of tiers to identify.

Recycling of rubber materials can be difficult since they are frequently constructed of complex polymers that are difficult to handle and break down. For instance, certain rubber goods contain synthetic components that cannot decompose and must be recycled using specific techniques [5].

Limited markets: The market for recycled rubber goods may be small, and demand for them may not always be great. Because of this, it may be difficult for rubber recyclers to find customers for their goods, which might result in an accumulation of trash and more expensive processing expenses.



Fig. 4 Dumping of tyre waste [5]

Energy-demanding: Recycling rubber uses a lot of energy, especially if it needs to be broken down and processed again into a useful state. If the recycling process is energy-intensive, it may be difficult to provide considerable environmental advantages. [4]

Costly: Rubber recycling may be cost-intensive since it requires a lot of work and expensive machinery to sift, shred, and process the rubber. Particularly for small-scale rubber recyclers, these expenses might be difficult to overcome.

Overall, rubber recycling may help decrease waste and save resources, but several difficulties and issues must be resolved for the process to be more successful and long-lasting [4].

A sieve analysis process developed to separate active rubber particles is used to examine the particle size distribution of rubber waste. This entails sorting the rubber waste into various size fractions using a succession of sieves with progressively smaller apertures. The particle

size distribution that results is then critically tested and contributes variously to the mechanical, thermal, and chemical properties of the reused active rubber particles.

When dealing with large amounts of rubber waste, the sieve analysis technique becomes time-consuming and labour-intensive. Furthermore, there is a possibility of affecting the mechanical properties and particle size distribution of active rubber particles throughout the analysis. To ensure the constant manufacture of high-quality particles, the sieve analysis technique for creating active rubber particles must be optimized. Efforts to optimize include selecting appropriate sieves, identifying the best sieving duration, and implementing methods that improve the accuracy and precision of particle size distribution data. This rigorous approach is required to achieve sustainability in the reuse of rubber waste and the production of commodities with characteristics.

To generate active rubber particles that may be utilized in a variety of applications, the present research attempts to optimize the sieve analysis process for rubber waste. Furthermore, it boosts the production of active rubber particles, decreases waste, and enhances sieve analysis process efficiency. It also increases the consistency and quality of the obtained active rubber particles using the sieve analysis technique.

The goal of this study is to analyse and evaluate the literature that already exists on rubber waste sieve analysis and then to find an optimum sieving procedure to get the maximum mass per cent of sieved active rubber particles. The critical variables that influence the sieving process, to obtain the best sieved active rubber particles are summarised as follows:

To design and carry out experiments to optimize the sieve analysis process with process variables like mesh size, sieving time [6], and particle size distribution of the rubber waste. Therefore, it analyses the experimental data and determines the best settings for the sieve analysis of rubber waste. Through frequent testing and comparison with the repeatedly obtained processes, the improved process conditions can be confirmed. Additionally, it provides the best procedures for producing homogenous, high-quality active rubber particles from rubber waste. Finally, based on the optimal findings it makes suggestions for potential uses for the active rubber particle, such as the production of rubber goods or as a filler for building supplies. [7]

I. THEORETICAL BACKGROUND

1. WHAT IS NATURAL RUBBER

Since the project is related to recycling waste tyres, which is otherwise a great problem, and since a significant weight per cent of the rubbery portion of the tyre is composed of natural rubber and finally, since natural rubber is overwhelmingly obtained from natural sources in the form of the natural rubber plant, it would not sound out of place to discuss and brief about natural rubber.

Rubber can be both natural and synthetic. Each, whether natural or synthetic, is an elastomer having rubber-like properties. Natural rubber is an elastomer made from a type of latex extracted from the bark of the *Heave brasiliensis* tree. [8] An illustration of tapping of the latex from the natural rubber tree is shown in Fig. 5.



Fig. 5 Natural rubber latex [9]

Natural rubber is made up of loosely coupled, tangled isoprene monomers that can be pulled apart but still return to their original shape when the force is released. This results in the acquisition of inherent elasticity and flexibility a characteristic feature of a rubber.

The manufactured products obtained from natural rubber are tyres, anti-vibration components, springs and bearings, rubber bands, adhesive materials, automotive parts, Industrial hoses, and latex products. Since natural rubber is durable and heat resistant, it is used in the production of high-performance tyres.

2. USES OF RECYCLED RUBBER

The world generates a large amount of rubber waste, mainly from old vehicle tyres. Rubber waste has been reduced by recycling and reusing these EOL tyres. Rubber from old vehicle tyres, shown in Fig. 7 can be broken down and recycled into products such as playground mulch, car components, mousepads, and shoe soles. [4]

After the rubber has been separated from the rest of the material, it is cleaned, and smaller pieces are ground up into even smaller fractions, depending the rubber may be further shredded into smaller fractions for more effective reprocessing, depending upon the demand for the quality of the reprocessed product. Material handling units, such as the high-energy transfer conveyor, are specifically designed to move this type of material and other tyre components, such as tyre wire. [10]

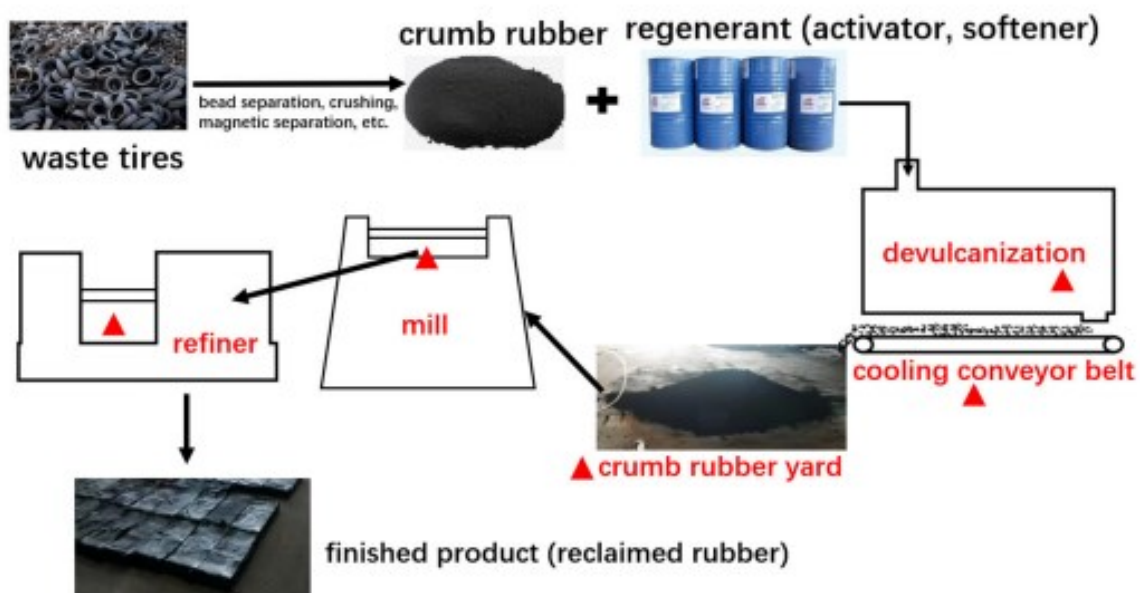


Fig. 6 Typical process flowchart of waste rubber tyre recycling [11]

|Types of rubber

In this chapter, various types of rubbers are briefly introduced:

Natural rubber is a particular type of elastomer; it is not a category. Although there is only one kind of natural rubber, it may be obtained from several plants and nations and modified to suit a variety of industrial needs.

Nitrile rubber is widely used and has strong fluid resistance, making it ideal for seal applications in manufacturing and the industrial sector. It is extremely resistant to alcohol, gasoline, and lubricants. Disposable gloves are one of the most typical uses for nitrile rubber. [12]

Ethylene propylene diene methylene rubber is kind of rubber, which can withstand heat, environmental damage, and ageing. Due to its durability and suitability for electrical applications, it is frequently utilized as a cheaper substitute for silicone.

Butyl rubber is kind of rubber, which is shock-absorbing and resistant to abrasion, heat, chemicals, and moisture. It is frequently utilized in sealants and electrical insulation. [13]

Hydrogenated nitrile rubber is more resistant to high temperatures, chemicals, hot water, steam, and fuels than regular nitrile rubber. Although it is extremely durable and resistant to abrasion, it cannot be used for electrical insulation or organic solvents.

Chloroprene rubber is rubber, sometimes known under its commercial name Neoprene, is flame- and oil-resistant. It is utilized for braces, some medical equipment, and coating textiles and seals. [14]

Epichlorohydrin rubber is resistant to acids and ozone, it should not be used for sealing applications since it could corrode metals and parts that are joined to metal.

Utilization of rubber in tyre manufacturing

Elastomers, strengthening additives, epoxies, and other ingredients are combined to make the rubber used in tyre manufacturing. The creation of tyres requires both natural and synthetic rubber. Trees that produce an abundance of milky white fluid made of rubber aggregates are the primary sources of natural rubber. While offering great rigidity, resistance, and consistency in compound compositions, natural rubber reduces intrinsic overheating in tyres.

The rubber used by the tyre business is synthetic to a 60 % extent. Petroleum compounds with a hydrocarbon basis serve as the precursor for their creation. Synthetic rubber deforms under pressure before resuming its original form. The creation of high-grip tyres with

significant traction benefits greatly from this trait. Additionally, synthetic rubber has distinctive qualities, particularly in terms of durability and inherent resistance. [15]

3. RECYCLING OF RUBBER (TYRES)

Tyres are the most common source of rubber that ends up as waste. To recycle the rubber, it first needs to be cleaned and then made into a smaller, more manageable size. The rubber is then placed into a granulator and transformed into crumb rubber small, fragmented pieces of rubber to be used elsewhere. [16] According to the European Tyre and Rubber Manufacturers' Association (ETRMA), the automobile industry accounts for around 65 % of the production of general rubber goods, including tyres, wiper blades, seals, hoses, seatbelts, gaskets, and insulators. Used tires are the most common source of waste rubber. According to the estimated figures, roughly 1,000 million tyres worldwide are unfit for reuse. [17]

The environment and public health are seriously threatened by the growing number of discarded tyres. Waste tyres that have been disposed of illegally pose a risk of uncontrolled combustion. Sulphur oxides, polycyclic aromatic hydrocarbons, small particles, and other toxic substances were released when scrap tyre rubber was burned (Fig. 7). [18]

As a result, populations living close to illegal tyre dumps and recycling rubber plants, particularly those built on prototype lines with weak foundations, are at higher risk of developing cancer. Furthermore, improper tyre disposal might result in rainwater pools that are stagnant due to the curved geometries of the tyres. These circumstances make it easier for harmful substances (such as heavy metals) to leak out of used rubber trash, which contaminates groundwater and harms aquatic life. [19] High humidity in abandoned tyres also fosters the growth of mosquitoes and rodents, both of which can spread disease.

Energy recovery is a popular practice nowadays for managing waste tyres, in which tyres or scrap tyres are used as an alternative fuel in power plants, paper mills, and cement kilns. According to the European Tyre and Rubber Manufacturers' Association ETRMA's most recent statistical report on the management of EOL tyres, approximately 91 % of waste tyres in the nations of the European Union, as well as Norway, Serbia, Switzerland, and Turkey, were properly collected and managed in 2018 through material recycling (56.4 %) and energy recovery (34.9 %). [20]



Fig. 7 Burning of discarded tyres

|Importance of recycling of rubber (tyres)

The formation of three-dimensional cross-linked structures in rubbers during vulcanization provides the performance properties of rubber goods (for example, tensile properties, abrasion resistance, chemical resistance, or thermal stability). Crosslinked rubbers, on the other hand, are non-biodegradable and cannot be easily recycled. [21]

The characteristics of rubber goods mentioned above make the management and recycling of post-production and post-consumer waste rubbers extremely difficult. The growing number of discarded tyres poses a serious threat to the natural environment and human health. Waste tyres that have been illegally dumped or stockpiled pose a risk of uncontrolled combustion.

Nowadays, energy recovery is a common method for waste tyre management, in which whole or scrap tyres are used as an alternative fuel in cement kilns, paper mills, or power plants. [22]

|Advantages of recycling rubber

There are numerous benefits of recycling rubber, with most of them linking back to the environment. Here are a few key benefits outlined below:

Reduces illegal dumping: Recycling rubber products can have a positive effect on the environment by reducing and discouraging illegal dumping of recycled rubber, as well as other products. If a mixture of products and wastes is illegally dumped, it could be harmful to the environment if the area is located near lakes or rivers.

Helps preserve the environment: In addition, reducing the demand and the production of new natural rubber through recycling can help preserve the natural resources usually used to create it.

|Disadvantages of recycled rubber

Although there are benefits to recycling rubber, recycling sites and the quality of the recycled rubber products also need to be considered and discussed.

Recycling sites: Depending on where your rubber is recycled, the process could be causing more harm to the environment than good. Some recycling sites with the sheer amount of waste they are collecting, are hosts for bacteria, disease, and other gases that could be harmful to water, air, and soil. Therefore, this means that recycling your rubber at some recycling sites could be worse for our environmental surroundings, than good. [23]

Compromised quality: Overall, the rubber recycling process is still being developed, and therefore recycled rubber products are generally seen as being of lower quality and having less durability compared to when their original form. As the demand for recycled rubber products is generally low on a global level, unfortunately there is not any rush for advancements to be made in the process that would change the quality and durability levels of rubber products. [24]

|Alternative uses for recycled rubber

There are many different uses for recycled rubber. A few outlets recycled in which waste rubbers can be used are as follows:

Astroturf, playground surfaces, non-slip mats, road surfaces and infrastructure, vehicle mats and interiors, hospital floors and gardening products and equipment. [25]

4. METHODOLOGY OF RECYCLING

|Grinding technologies for waste tyre rubbers

The basic separation of waste tyres includes two streams: passenger car tyres and truck tyres. Off-road tyres are sometimes considered a third stream of waste tyres. This is because the composition of these three types of tyres differs significantly. [26]

In comparison to passenger car tyres, truck tyres have a higher content of natural rubber and a lower content of carbon black as reinforcing filler. These differences can be attributed to higher performance characteristics of passenger car tyres, such as low rolling resistance, improved skid resistance, and long wear. [27] Off-road tyres have a higher content of textiles (10 % by mass) than passenger tyres (5.5 % by mass) or truck tyres (1 % by mass), which may cause problems with the purity of ground tyre rubber based on off-road tyres. [28]

Ground rubber has different particle sizes, surface characteristics, and the like, depending on the grinding technology and conditions. Steel, textile cord, and ground tyre rubber are the three streams of waste tyre-grinding products. [29]

According to the Web of Science, the most common term “crumb rubber” is used in the following fields: engineering civil (1,036 items); materials science multidisciplinary (990 items); and construction building technology (873 items) [30]. According to the Scopus database, most works with the term “crumb rubber” are related to engineering (5,595 items) [31], materials science (3,582 items), and environmental science (1,262 items) [29]. These data show that the focus of research work is on the use of waste rubbers as environmentally friendly materials in civil engineering, construction and building applications, and material science (the automotive industry is the most desired target). [31]

The term ground tyre rubber appears to be the best option for uniform nomenclature because it indicates the source of waste rubber as well as the final form of material obtained after shredding and grinding [32]. Alternative terms include waste tyre rubber and waste tyre dust. Furthermore, it should be noted that the term “tire” was used as a form in American English during the search, whereas results for the term “tyre” in British English may differ. [33]

The two most common industrial-scale waste tyre grinding technologies are ambient and cryogenic. The first method involves passing waste rubber through shearing mills or two-roll mills that operate at room temperature [34]. However, when waste rubber is ground at ambient

temperature, the temperature of the ground rubber can rise to 130 °C [35]. This phenomenon raises the possibility of uncontrolled rubber combustion during grinding. Cryogenic waste tyre grinding is based on effectively cooling waste rubbers below their glass transition temperature using liquid nitrogen. This converts the elastic rubber material into a frozen brittle form that can be easily crushed with a hammer mill. [36]

Different mechanisms of breaking during waste tyre rubber disintegration by both methods affect the surface and shapes of ground tyre rubber [37]. Ground tyre rubber is characterized by an irregular particle shape and a spongy, well-developed surface in the ambient method, whereas in the cryogenic method, rubber particles are regular and smooth, with a low surface area [38].

Other waste rubber grinding methods, such as grinding with the help of ultrasounds, cutting with water under pressure (waterjet), pulverization in the presence of supercritical carbon dioxide, and other prototypes, are gaining popularity [39].

Ultrasounds are used to activate rubber grinding technology. This method enables the production of ground rubber with a very good particle size distribution [40] and average particle sizes in the range of 150 μm , which can be used to replace reclaimed rubber in the industry [41]. Furthermore, the results demonstrated that using ultrasounds during waste rubber grinding reduces energy consumption while increasing production efficiency through faster shredding speeds [42].

Recently, there has been a surge in interest in the development of waste rubber pulverization/grinding technologies, as well as their appropriate treatment via devulcanization or functionalization. investigated the cryogenic grinding of ground tyre rubber modified by supercritical carbon dioxide (scCO_2) [43] devulcanization in a fluidized-bed jet mill. The effects of varying scCO_2 content on ground tyre rubber devulcanization and grinding efficiency were investigated. Supercritical fluids (scCO_2 , ethanol) and subcritical fluids (water) can swell waste rubber, which improves devulcanization by increasing the selectivity of cross-linking bond-breaking [44].

Rubber reclaiming, in conjunction with devulcanization (de-crosslinking), is one of the oldest and most popular methods for treating ground tyre rubber, which has developed successfully in recent years, as summarized in comprehensive review works. This method allows the transformation of cross-linked rubber using thermal, mechanical, or chemical energy into reclaimed rubbers, which can be easily reprocessed, shaped, and vulcanized. [45]

Devulcanization allows for the highly selective breaking of sulphide cross-linking bonds, whereas rubber reclaiming also results in the scission of the main chain. Increasing the level of main chain scission resulted in a decrease in polymer molecular weight and an increase in polydispersity, both of which typically degrade the mechanical properties of vulcanized reclaimed rubbers, or vulcanizates. [45]

Differences between reclaiming and devulcanization based solely on subjective assessment and analysis of the main chain degradation degree are usually supported by Horikx theory [46]. However, it should be noted that swelling/treatment conditions (e.g. temperature, time, sample weight, type of solvent used) will have an impact on the values of parameters used for estimating main chain scission selectivity based on Horikx theory [47].

Recent trends in waste rubber treatment technology research have shown that low-temperature devulcanization and the combination of devulcanization with appropriate functionalization are gaining attraction [48].

|Challenges and future trends related to ground tyre rubber

Many attempts in waste tyre recycling have been depicted over the last ten years, particularly those focused on the direct application of ground tyre rubber in various matrices (e.g. polymers, bitumen, concretes, etc.) or ground tyre rubber devulcanization treatment.

Current advances in waste tyre rubber grinding technologies and treatment methods, which allow tailoring of particle size distribution and suitable modification/functionalization of GTR surface by reactive functional groups or physical encapsulation by compatibilizers, received special attention [49].

5. TECHNIQUES FOR RECYCLING THE RUBBER

Rubber is a durable and versatile material that, due to its non-biodegradability, presents a significant environmental challenge. To address this problem, several recycling techniques have been created to reduce the environmental impact of rubber waste. Among the most popular methods for recycling rubber are mechanical grinding, vulcanization, and devulcanization of reclaimed rubber.

Mechanical grinding

Mechanical grinding of rubber includes the use of mechanical forces to break down rubber waste into smaller particles. This method is commonly used in rubber recycling to convert waste rubber into reusable material. [50]

Initially, the rubber waste is fed into a shredder, which cuts it into smaller fragments. These pieces are then fed into a grinding machine, which employs rotating blades to further reduce the size of the rubber particles. Other forces, such as compression or impact, may be used during the grinding procedure to improve the breakdown. The resulting rubber particles are used for a variety of purposes, including the production of mats, outdoor equipment, and tyres. By changing the grinding machine's parameters, the size of the particles can be managed. [51]

When compared to other rubber recovery methods, the mechanical grinding of rubber has several benefits. It is a simple, economical process that is simple to scale up to handle big amounts of rubber waste. The method is adaptable because a variety of uses can make use of the rubber particles that are produced. The disadvantages of motorized grinding do exist. High amounts of noise and dust can be produced during the process, which could be hazardous to workers' health. Additionally, the heat generated during grinding may result in the degradation of the rubber particles, adversely impacting their properties. It is crucial to use the right safety precautions and equipment when grinding to reduce these dangers. [52]

In conclusion, mechanical rubber grinding is a recycling process that uses mechanical pressures to reduce rubber waste into smaller particles. This method has several benefits, including affordability and adaptability. However, it also has some drawbacks that need to be addressed, such as possible health risks and rubber particle degradation because of heat.

Vulcanization and devulcanization

Vulcanization is a chemical process that enhances the qualities of rubber, making it more durable, elastic, and resistant to various environmental influences. This procedure, pioneered by Charles Goodyear in the nineteenth century, involves heating raw rubber with sulphur or other chemicals to cause cross-linking of the polymer chains. This cross-linking process forms a three-dimensional network within the rubber, increasing its strength, flexibility, and heat resistance. Vulcanization converts rubber from a soft, sticky material into a flexible substance suited for a wide range of applications, including tyres and industrial seals. It has a significant impact on modern business and daily life, ensuring the dependability and lifespan of rubber products across several areas.

Devulcanization stops this process by breaking down the sulphur bonds and restoring the elasticity of the rubber. Devulcanized rubber can then be used to create new rubber goods. [53]

Devulcanization of rubber refers to the process of breaking down the cross-links created between rubber molecules during vulcanization. This process is critical in rubber recycling because it enables the reuse of waste rubber that would otherwise be discarded. Devulcanization can be done using a variety of methods, including chemical, thermal, and mechanical techniques. Chemical devulcanization is the process of using chemicals to break down the cross-links between rubber molecules. Thermal devulcanization involves the use of heat to break down the cross-links, whereas mechanical devulcanization involves the use of mechanical pressures to break down the rubber molecules. [54]

Once devulcanized, the rubber can be reused for a variety of uses, including the manufacture of new rubber products. Many of the properties of virgin rubber, such as elasticity and strength, are retained in devulcanized rubber, making it a valuable resource for the rubber business. However, the devulcanization procedure is not without difficulties. One of the most difficult challenges is achieving a high degree of devulcanization while retaining the desired rubber properties. Over-devulcanization reduces the strength and elasticity of the rubber, while under-devulcanization reduces efficiency and durability.

Devulcanization has advanced significantly in recent years thanks to the creation of new technologies and methods by researchers who wanted to increase the process' efficacy and efficiency. Devulcanization of rubber is turning into a more significant field of research and development as the demand for environmentally friendly and sustainable solutions keeps rising [55].

|Reclaimed rubber

This method entails removing rubber from abandoned items like tyres and refining it to create new rubber goods. To improve its qualities and suitability for various uses, reclaimed rubber is frequently combined with other materials, such as synthetic rubber or carbon black. [56]

Reclaimed rubber is a rubber that has been processed after it has fulfilled its original purpose of removing impurities and improving its usability. It is a more environmentally friendly and cost-effective option than virgin rubber, and it is used in a variety of applications such as tyre manufacturing, automotive parts, footwear, and construction. Rubber reclaiming entails shredding waste rubber into tiny pieces and subjecting it to several treatments, including chemical and mechanical processes, to remove impurities and break down the rubber molecules. To accomplish the required properties, the resulting material is then blended with virgin rubber or other additives. [57]

Reclaimed rubber has several benefits over virgin rubber, including lower costs, less environmental impact, and less reliance on non-renewable resources. It also has properties that make it ideal for specific uses, such as increasing the elasticity and durability of rubber products. However, the use of recycled rubber has some restrictions as well. It might not have the same qualities as fresh rubber, and it might not be appropriate for all uses. Additionally, the procedure for recovering rubber can be time-consuming and necessitates specialized tools and knowledge. [58]

In general, using recycled rubber is a practical and affordable way to reduce waste and advance environmental sustainability in the rubber business. The use of recycled rubber is anticipated to increase in popularity across a variety of uses as consumer demand for green materials rises. [59]

|Sieving process

The rubber sieving process, which involves the separation of rubber particles of varying sizes through a sieve or mesh, is an important step in the rubber industry. The importance of this process stems from the fact that rubber particles of varying sizes have distinct properties and are used in a variety of applications. [60]

The first step in the procedure is washing the raw rubber material to remove impurities like dirt. After being thoroughly cleaned, the rubber material is fed into a device with several screens or sieves of various sizes (Fig. 8). Rubber particles travel through the various screens as they are separated by size thanks to the machine's vibration of the sieves [61].



Fig. 8 Sieve Analyser

Large rubber particles that cannot travel through the first screen are gathered and brought back to the machine for additional processing. The rubber particles that effectively pass through the first screen, on the other hand, are collected and passed through the next screen, and so on until the rubber particles are separated into the desired sizes.

The rubber particles are dried and packaged for shipment or additional processing after the sieving process is finished. High-quality rubber goods must be produced using the sieving method, which guarantees that the rubber particles are uniform in size and free of impurities. [62]

II. EXPERIMENTAL PART

6. PURPOSE OF THE WORK

Due to its vast volumes and sluggish disintegration rates, waste rubber is a serious environmental issue, but it also represents a priceless resource that may be recycled and applied in several industrial settings.

The scope of the present work framing the thesis is to examine the waste rubber particle size distribution using sieve analysis which is important to assess any potential industrial uses.

Thus, a general conclusion to the objective of the work can be written as follows.

Waste tyre rubber particle size distribution significantly influences its characteristics and appropriateness for various industrial uses. Understanding the waste tyre rubber particle size distribution can help one improve its characteristics and create new uses for this useful commodity.

Particle size distribution of scrap tyre rubber may have industrial uses in the creation of building materials, asphalt binders, and sound-absorbing materials, which will be explored in this thesis. The findings of this thesis can be applied to create guidelines for waste tyre rubber particle size distribution optimization in a variety of applications, resulting in enhanced product performance and cost-effectiveness.

Based upon the scope and the objectives of the work, practical experiments were designed to meet the same. They may broadly be divided into three categories:

- Sieve analysis to separate the various mass fractions.
- Remedial treatments such as drying and solvent treatment followed by drying of the original powdered particles.
- Characterization of the received and the variously treated powdered particles to develop a comprehensive idea about the changed condition of the particles and to assess the best treatment method, suitable for the fitness of the purpose in the direction of obtaining the maximum mass fraction below a defined sieve dimension.

7. MATERIAL

The waste ground active rubber particles were received from an industry in which there was a mixed sample of different types of rubber waste, some old used tiers and some newly produced tiers but rejected for industrial quality protocol. Fig. 10. According to the industry, the already ground-used tiers and the size of the particles are less than 500 μm . However, when sieved, it was found that some residues were above 500 μm .



Fig. 9 Ground rubber sample

8. METHODS

Sieving of the originally obtained rubber particles using sieves of retention dimensions of 500 μm , 400 μm , and 300 μm and a base plate to collect particles lesser than 300 μm . Repetitions were done to optimize the process variables such as the time of sieving and the initial mass of the rubber particles to be taken for the sieving. The optimization of the sieving process is now discussed in detail.

Sieving process

8.1.1. Optimization of time of sieving

According to the experiment, it is important to find the optimal conditions of sieving for standardizing the process to obtain the best results.

Accurate Particle Size: The sample is effectively and efficiently sieved, producing a more accurate and representative particle size distribution. This is ensured by selecting the best conditions and timing for sieve analysis. When the performance of the substance being studied depends on its particle size distribution, this is very essential.

Consistency: For sieve analysis to be consistent and repeatable, the right conditions and amount of time must be used. This is crucial for quality control purposes. This makes it possible for different operators to get the same outcomes consistently over time.

The optimal condition also provides the important behaviour of the material being analysed it can help to solve some problems, such as agglomeration and particle breakage, and it also helps to improve the quality of the analysed material.

In this research, to find the optimal time conditions, sieve analysis was performed seven times with different durations: 20 min, then 40 min, 60 min, 80 min, 100 min, 120 min, and 140 min. This process was repeated twice.

8.1.2. Optimization of the weight of the initial sample for sieving

For the optimal weight of the sample, two runs were performed with samples of different weights (50 g, 100 g, and 200 g), each at the optimized time of sieving. The major goal was to

find the weight that produced the best sieving results in terms of particle separation and consistency.

8.1.3. Sample collection for modification

In total, fourteen runs, each comprising 200 g of the original rubber powder, were sieved at the optimal conditions, and the powders obtained from the sieving below 400 μm were collected for further treatments, such as drying and solvent treatment, the details of which had already been discussed.

Three different types of sieves were utilized with mesh sizes of 500 μm , 400 μm , 300 μm , and dish (Fig. 10).

A sample of 200 g of ground active rubber particles was collected to conduct a sieve analysis. Once the rubber sample had been weighed, a series of sieves with progressively smaller mesh sizes were employed to separate it. On the top sieve of the stack, the 200 g rubber sample was put for 60 min, the stack of sieves was mechanically shaken vigorously enough at 1.5 amplitudes to allow the rubber particles to pass through the sieves following the shaking, and the number of rubber particles still on each sieve was weighed.

The particle size distribution of the rubber sample was then determined using the weights of the retained rubber particles. The industry claimed to have sent the sample in a size below 500 μm . After performing the process, some of the particles still didn't sieve from the 500 μm mesh; however, for this project, the sample size should be below 400 μm for different industrial applications.

The collected sample, which was below 400 μm , was re-sieved, and still, there were some rubber particles collected in the 500 μm mesh. To find out the reason behind it, relevant literature was reviewed, and found that the possibilities for the generation of agglomeration were due to some wax present in rubber waste, or the sample was a mixed waste of different kinds of tyres.

To avoid the generation of agglomerate, the sample needed to be treated, and for that, a similar process was performed fourteen more times to collect the sample for further analysis. Overall, 2.6 kg of rubber particles were collected from sieving.

Further, the sample was collected and modified by treating it with acetone to reduce the wax content in the sample, and the other treatment was to dry the sample before sieving.

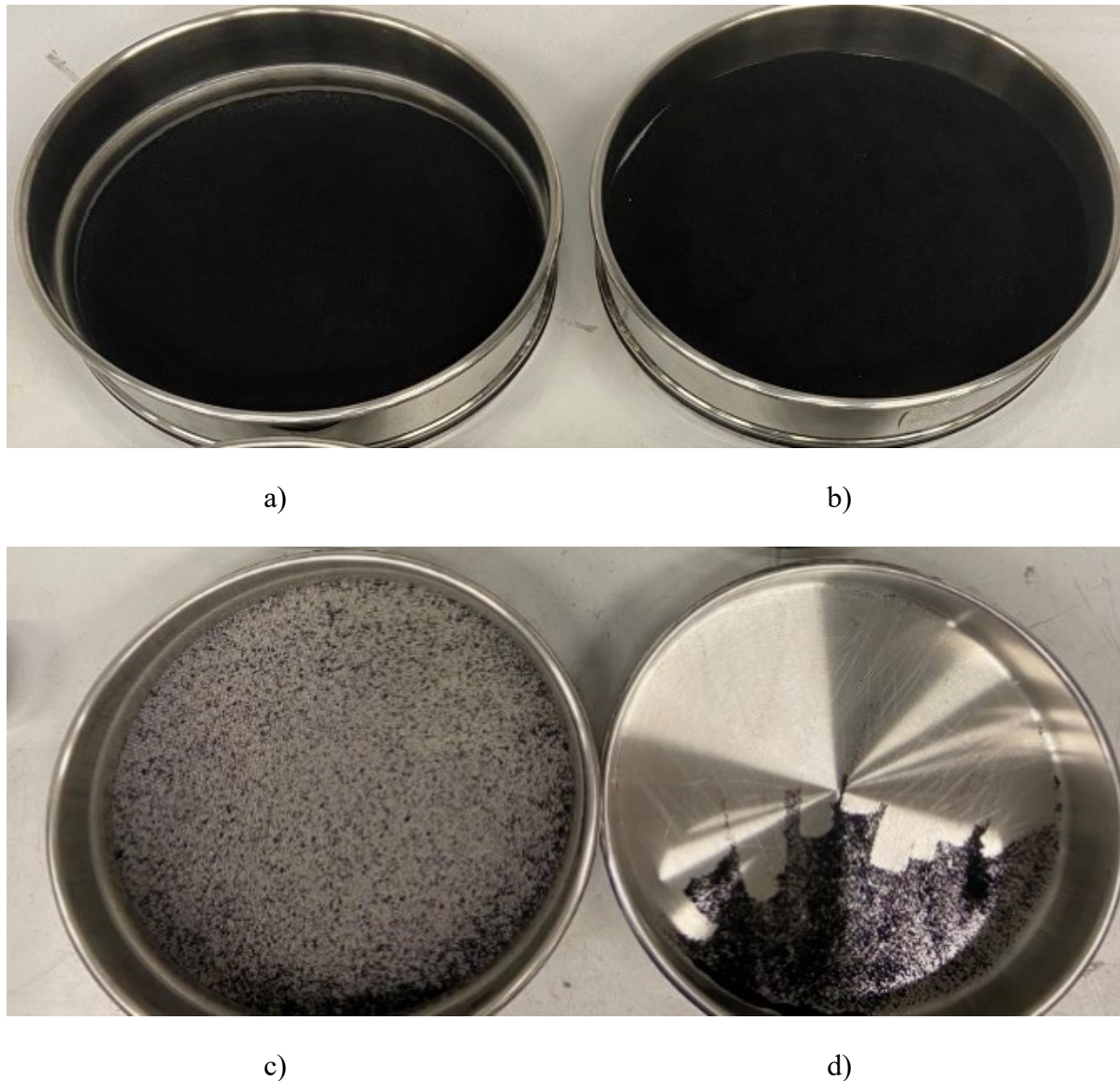


Fig. 10 Different sieves with corresponding fractions: a) 500 μm , b) 400 μm , c) 300 μm , and d) dish

Drying process

Drying grounded rubber is an important stage in the processing of recycled rubber. The drying process removes moisture and volatile chemicals, ensuring that the rubber has acceptable physical and mechanical qualities for future applications. Drying at 80 °C for 12 h

is a standard procedure that strikes a balance between effective moisture removal and the rubber material's integrity.

The drying process was carried out using a drying oven or a specialist industrial drier. Key equipment includes:

Oven: Can maintain a steady temperature of 80 °C. Trays and racks are perforated to promote uniform air circulation around the rubber particles.

Air Circulation System: Provides even heat distribution throughout the drying chamber.

Cleaning: The drying trays or racks were cleaned to avoid contamination.

Loading: The ground rubber was spread evenly throughout the trays to prevent clumping promote even drying and allow adequate air circulation. (Fig. 11).

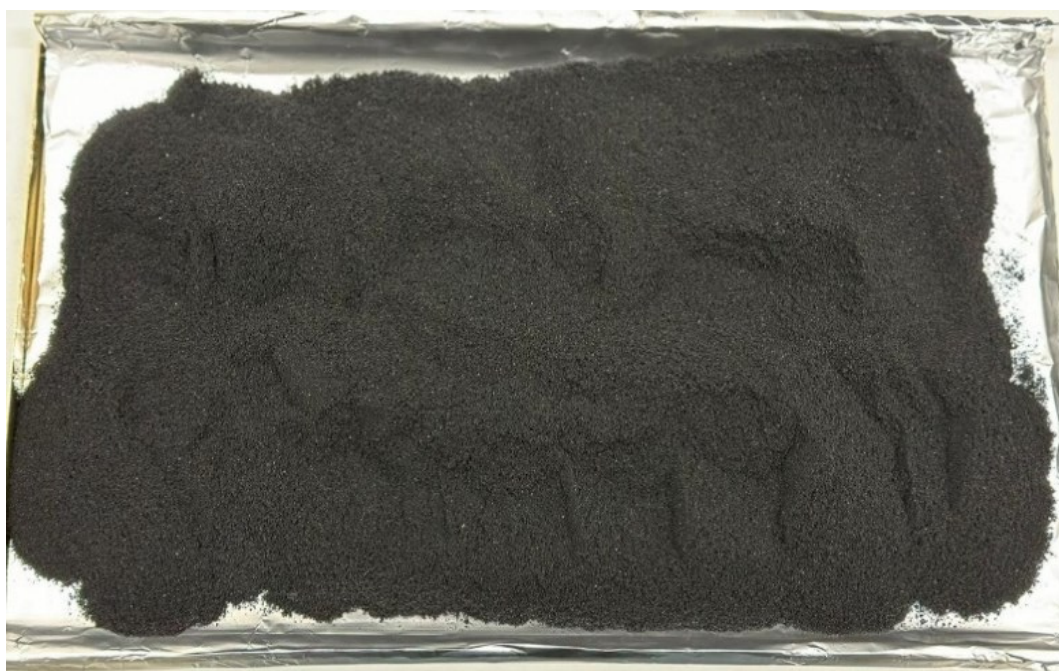


Fig. 11 Sample prepared for drying

Dry Time: 12 h of drying time was set according to experimental conditions for moisture and volatiles to escape from the rubber particles (Fig. 12).

After 12 h of drying, the oven was automatically switched off and the rubber cooled before handling. Rapid cooling can lead to condensation, which may reintroduce moisture.



Fig. 12 Oven condition for drying

Inspection of the rubber for dryness was done to avoid reabsorbing moisture from the environment, the rubber sample was stored in an air-tight bag.

Drying ground rubber at 80 °C for 12 h effectively removed moisture and volatile chemicals. This technique, when done correctly, increases the quality and performance of recycled rubber, making it ideal for a variety of industrial uses. Attention to detail in preparation, constant temperature regulation, and careful post-drying handling are critical to attaining the best results.

After treatment and drying, the sieving was performed again with the same standard 60 min at 1.5 amplitude, showing the result of sieving after treatment.

Acetone treatment

Acetone treatment is a common method in the rubber industry for extracting additives, wax, and some volatile materials.

A clean, dried sample of rubber was weighed and placed into a beaker, then acetone was added to the beaker according to the standard of the test, and then the beaker was covered to prevent evaporation (Fig. 13). The beaker containing the rubber and acetone mixture is placed on a mechanical shaker to mix the solution properly for 30 min to 60 min. After the treatment was complete, the mixture was filtered through a filter paper to separate the rubber from the acetone solution.

The residue was also rinsed with fresh acetone to remove the remaining impurities. The sample was further washed with water and dried according to the condition. For this research,

acetone treatment was performed to remove the volatile material from the surface of the rubber powders and to check the difference between raw sieved particles and extracted sieve particles.

750 g sieved sample was taken for acetone treatment from a collected sample with size 0–399 μm (Fig. 14).



Fig. 13 Extracted acetone sample



Fig. 14 Extracted sample for drying

The sample was left for 24 h to settle down the particle size. After 24 h, the acetone was decanted and washed with water to remove the remaining impurities. Then, the sample was dried in an oven at a temperature of 80 °C for 24 h (Fig. 15).

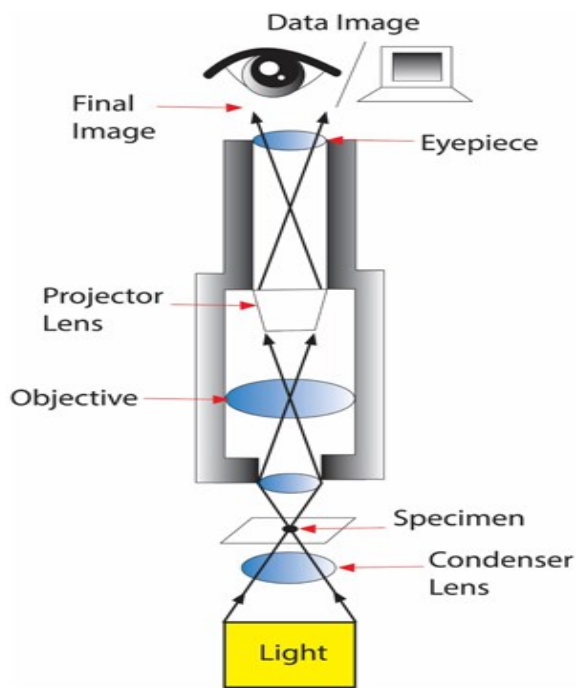


Fig. 15 Drying conditions after acetone treatment

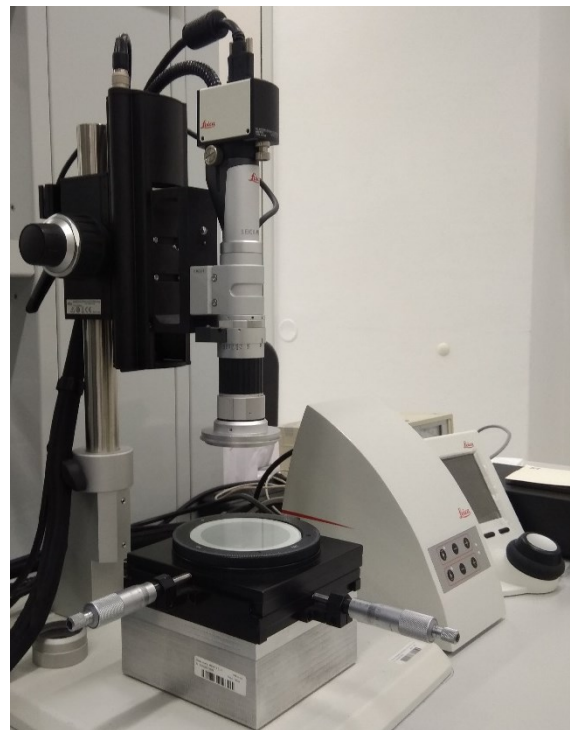
9. CHARACTERIZATION OF RUBBER PARTICLES

Optical microscopy

An objective lens, ocular lens, lens tube, stage, and reflector make up the bulk of a typical optical microscope. The objective lens will magnify an item put on the specimen stage. An enlarged picture can be seen through the eyepiece or on a computer screen after the target is focused. (Fig. 16) The objective and ocular lenses of the microscope will shine light onto or through objects (the sample set up on the specimen stage), magnifying the transmitted or reflected light.



a)



b)

Fig. 16 Optical microscope: a) Principle [63] and b) LeicaDVM-2500

The optical microscopy images of the fraction of rubber samples according to their sizes after sieving were captured by the digital optical microscope Leica DVM-2500 (Fig. 16).

|Thermogravimetric analysis

Thermogravimetric analysis, often known as TG analysis, is a thermal analysis technique that frequently records changes in a substance's weight as a function of time or temperature. TGA can measure a variety of properties and behaviours, including composition, purity, decomposition procedures, decomposition temperatures, and absorbed moisture content.

One of the greatest ways to ascertain the thermal properties of many materials, including chemicals, medicines, plastics, elastomers, and thermosets, as well as mineral compounds and ceramics, is through TG analysis.

TG analysis provides a quantitative assessment of any weight changes brought on by thermally induced transition. The procedure includes gradually raising the temperature of a sample, which is a material with a known beginning weight. At various time intervals, the weight changes are recorded as a function of temperature.

As a TG curve in which the weight change is recorded as a function of temperature or time. As a TG derivative TG, where the TG curve's first derivative is plotted against either temperature or time. The graph plotted is known as the pyrolysis curve.

TG analysis provides quantitative information about the content of the low volatiles (e.g. oil, wax, plasticizers, extenders, and the like), polymer(s), carbon black, and inorganic substances. TG analysis was performed on Thermo-Gravimetric Analyser TA Q500 (TA Instruments, USA, Fig. 17) in a temperature range from approx. +27 °C – +650 °C in nitrogen atmosphere followed by switching the atmosphere to air up to +850 °C. Samples of approx. 10 mg were taken from each run. Three runs were performed.

|Infrared spectroscopy

To measure the changes in a wholly internally reflected infrared beam that take place when the beam encounters a sample, an attenuated total reflection (ATR) accessory is used (shown in Fig. 18). An optically dense crystal with a high refractive index is exposed to an infrared beam at a certain angle. This internal reflection generates an evanescent wave that penetrates the sample kept in touch with the crystal as well as the crystal's surface. It may be simpler to picture this vanishing wave as an infrared bubble resting on the crystal's surface. Only a few microns (0.5–5 µm) of this evanescent wave extend past the crystal surface and into the sample. As a result, the sample and crystal surface need to make excellent contact. The evanescent

wave will be attenuated or changed in the infrared spectrum where the sample absorbs energy. Each evanescent wave's attenuated energy is transferred back to the infrared (IR) beam, which leaves the crystal's other end and travels to the IR spectrometer's detector. After then, the system produces an infrared spectrum.

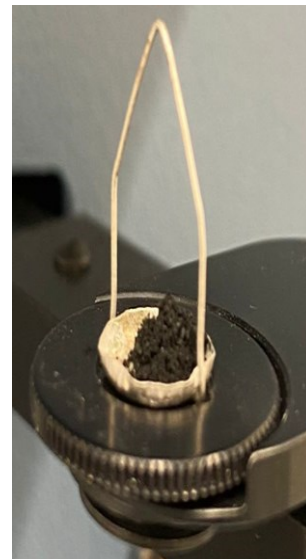
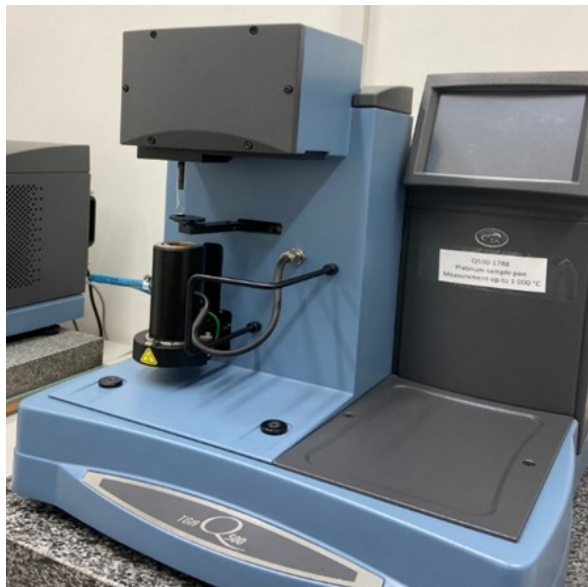
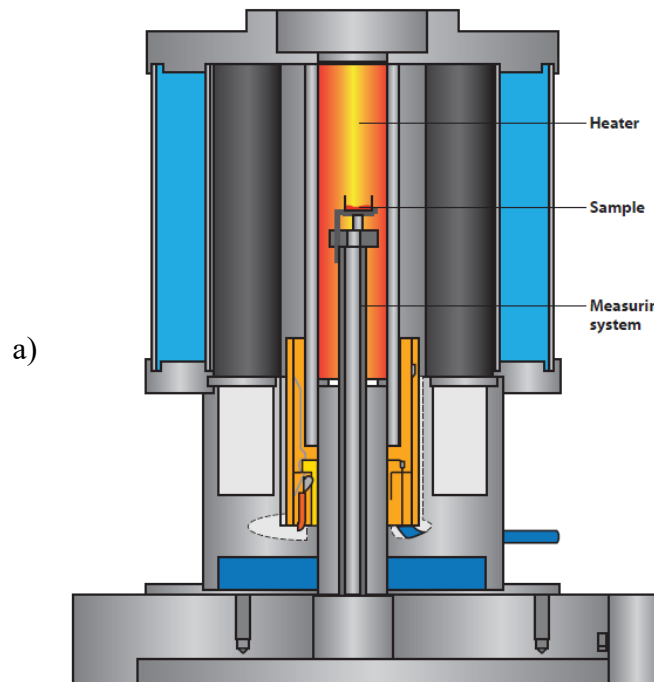
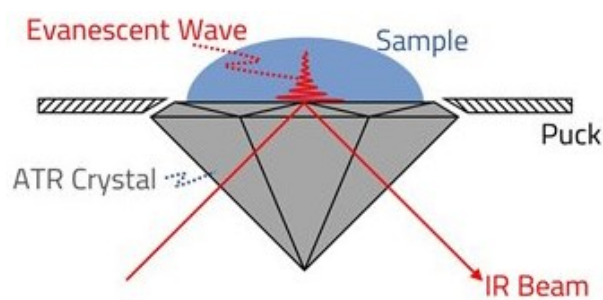
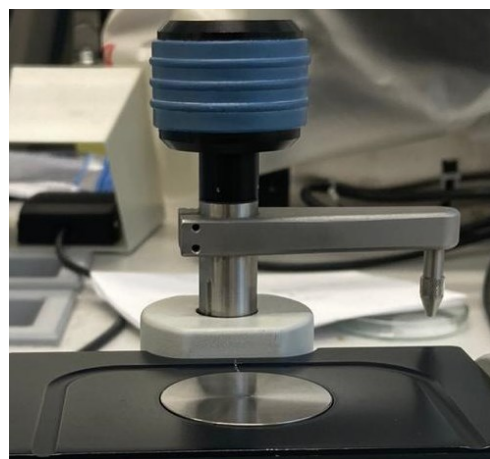


Fig. 17 TG analysis: a) Principle [64], b) TG analyser Q500-1788, and c) detail of sample holder and analysed sample



a)



b)

Fig. 18 IR spectroscopy: a) Principle [65], and b) detail of ATR cell with germanium crystal

The evanescent wave or bubble only extends 0.5 to 5 μm beyond the ATR crystal; hence the sample must be in intimate touch with it. Internal reflectance won't happen unless the crystal's refractive index is much higher than that of the sample; in this case, light will be transmitted through the crystal rather than internally reflected. Refractive indices for ATR crystals typically range from 2.4 to 4.0 at $2,000\text{ cm}^{-1}$. It is reasonable to infer that most organic liquids and solids have substantially lower refractive indices. Recent investigations have introduced ATR for inorganic work. Different crystals (such as germanium, ZnSe, and Diamond) must be used to get the best findings from some experiments.

ATR Fourier transform IR spectroscopic studies of the provided samples were carried out at a resolution of 2 cm^{-1} using a germanium crystal and the results obtained in the wavenumber range of $4500\text{ to }250\text{ cm}^{-1}$ (from the original data as obtained from the IR instrument, and also baseline subtracted by using a particular algorithm of baseline subtraction).

10. RESULTS AND DISCUSSION

Optimization for the time of sieving

Figs. 19 and 20. represent the two runs, each of which shows the different time allowed for the completion of the sieving process, ranging from a minimum of 20 min to a maximum of 140 min at regular intervals of 20 min, where the plot of percentage fraction retention versus the mesh size have been plotted.

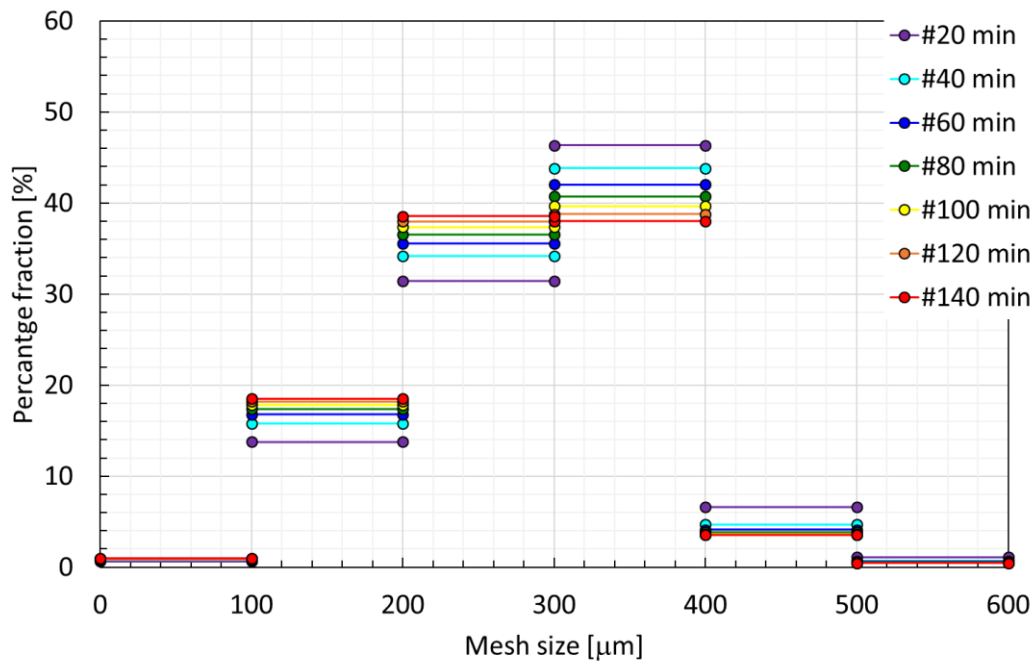


Fig. 19 Percentage fraction of original rubber particles for various times of sieve analysis for run 1

Time of sieving plays the role as shown. From both the runs of sieved particles, it was clear that the particle size of the material was 500 $\mu\text{m}+$. To resolve this problem, the important step was to optimize the time for sieving after sieving at different times the fraction percentage varied according to different times and sizes of the particles for 20 min the lowest amount of sieved particles above 400 μm and also the lowest amount of sieved particle below 100 μm as compared to other times. According to Fig. 21, where the fraction 500 $\mu\text{m}+$ is presented, the optimal sieving time is 60 min.

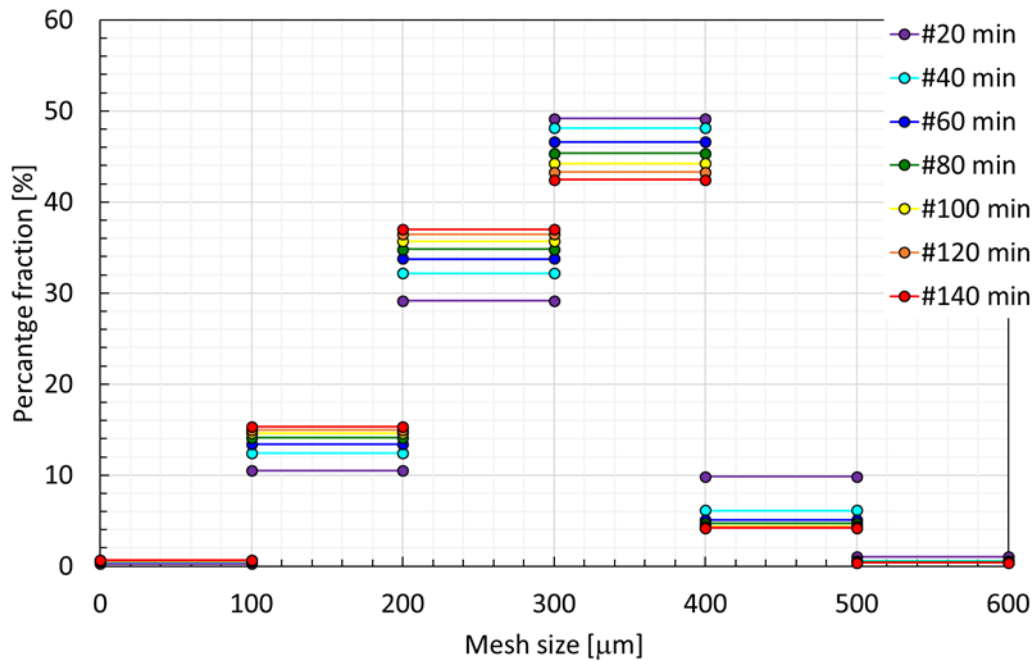


Fig. 20 Percentage fraction of original rubber particles for various times of sieve analysis run 2.

Efficiency Plateau: After 60 min, most particle size categories approach a plateau, suggesting that adding screening time does not appreciably increase the number of sieved particles.

Energy and Time Savings: Beyond 60 min, the incremental increase in sieved particles does not justify the increased time and energy used.

Uniform Sieving: After 60 min, a balanced number of particles of various sizes are successfully sieved, resulting in optimal separation.

This analysis aids in determining the most efficient sieving duration, maximizing production while minimizing time and energy usage. After the sieving, based on the result, 60 min is the optimal time for sieving the sample.

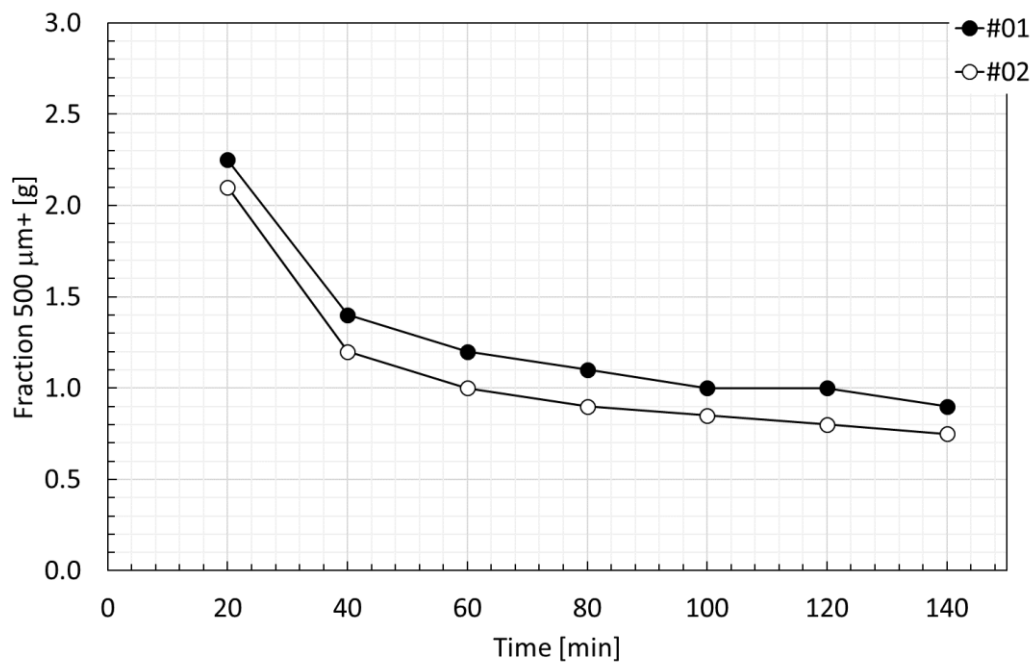


Fig. 21 Fraction of 500 µm+ collected at different times of sieve analysis

Optimization the mass of the rubber powder to be taken for sieving

Figs. 22 and 23 represent the two runs, each of which shows for the optimized of 60 min, three different masses of 50 g, 100 g, and 200 g, where the plot of percentage fraction retention versus the mesh size has been plotted.

The results showed that the 200 g sample produced the best results. This weight permitted a more thorough sifting process, resulting in a higher proportion of particles being categorized accurately based on size. The additional weight resulted in a more effective transmission of energy from the sieving device, improving particle movement and separation across the sieve mesh. As a result, the 200 g sample had a more complete spectrum of particle sizes, producing more precise and dependable results. In comparison, the 50 g sample showed less effective sieving. The lesser weight resulted in insufficient energy transfer, which limited particle movement and interaction with the sieve mesh. This resulted in increased particle retention on the early sieves and a less obvious separation of different-size fractions. Similarly, the 150 g sample outperformed the 50 g sample, although not as well as the 200 g sample. Although it improved particle mobility and separation, it fell short of the ideal distribution observed with the 200 g sample.

The findings highlight the importance of adjusting material weight in sieving analysis. The 200 g sample showed that a heavier weight might greatly improve the efficiency and accuracy of the sieving operation.

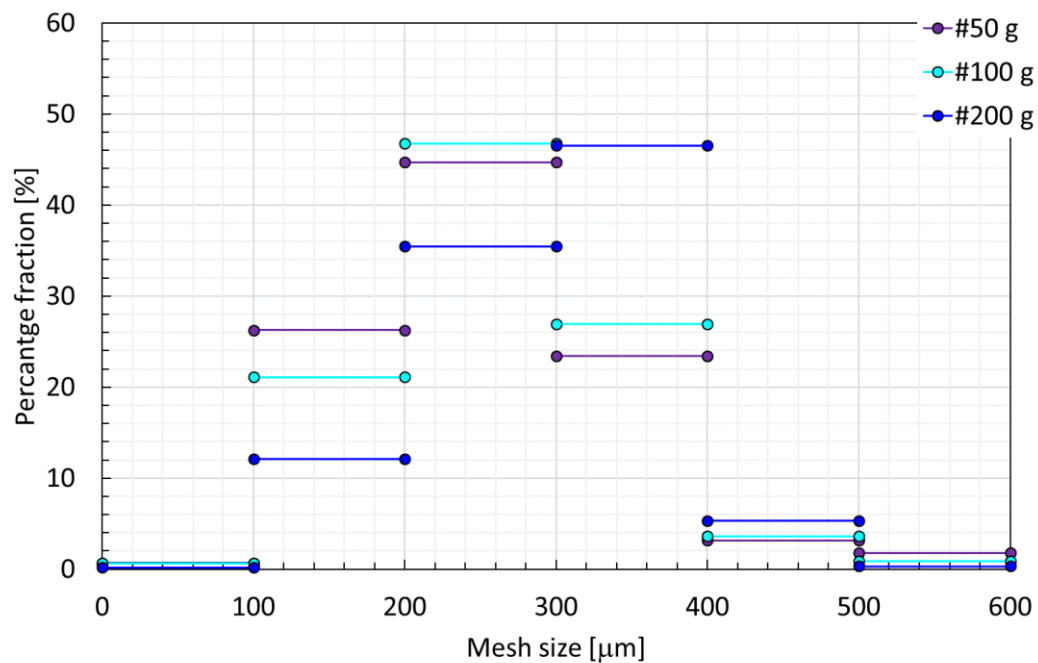


Fig. 22 Percentage fraction of original rubber particles for amount of sieved material for run 1

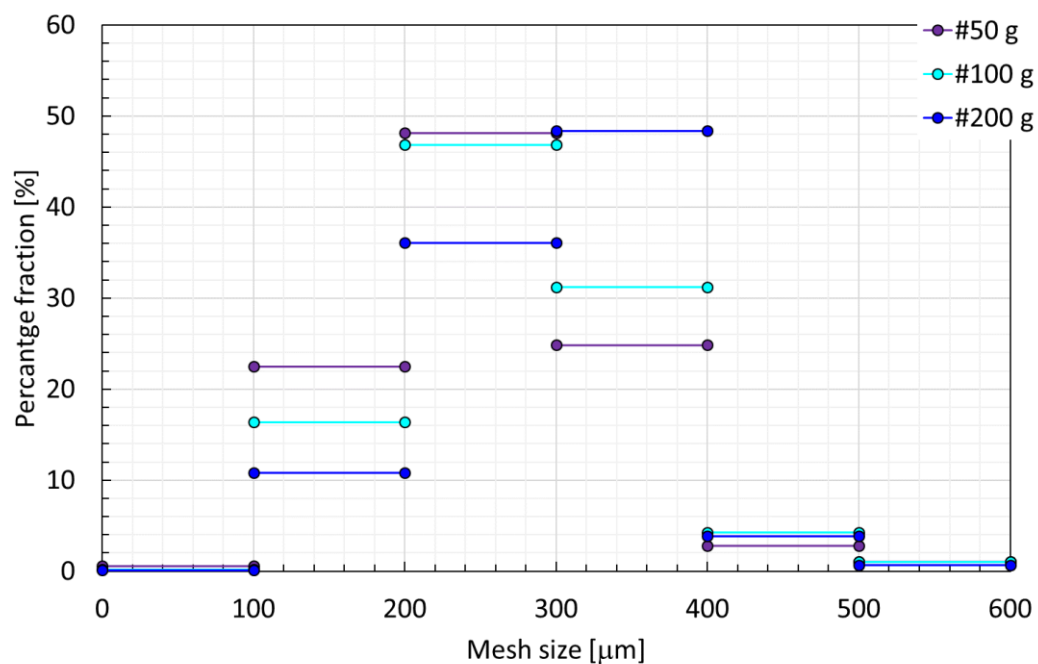


Fig. 23 Percentage fraction of original rubber particles for amount of sieved material for run 2

Sieving of the original particles under the optimized conditions

Once the optimization of time at 60 min and the mass at 200 g was obtained, three sieving runs at these optimized conditions were performed. The results obtained are shown in Fig. 24 showing the percentage fraction of the retained particles versus the mesh size of the sieves. Since the main interested was focused on the rubber particles 500 μm +, the sieves 500 μm , 400 μm , and dish were used for collecting of rubber particles.

Again, 500 μm + particles were found after completing runs each run shows a significant percentage of the particles left shown in the Table 1.

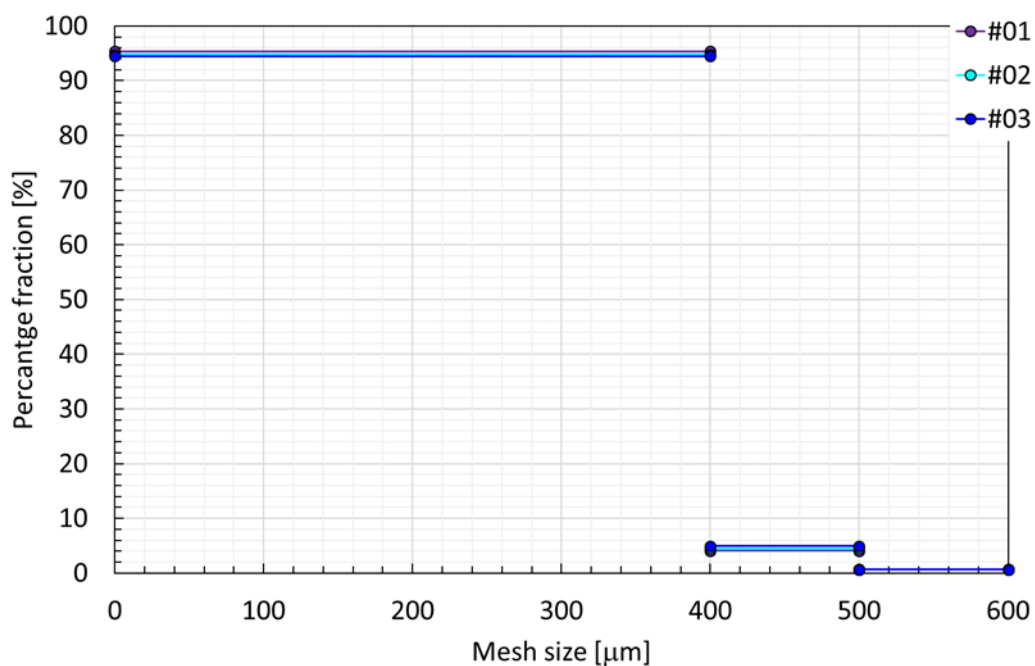


Fig. 24 Percentage fraction of original rubber particles obtained under optimized conditions

Table 1 Percentage fraction of rubber particles at different runs

Mess size [μm]	Run#01 [%]	Run#02 [%]	Run#03 [%]
500+	0.60	0.70	0.62
400–499	4.05	4.49	4.93

To monitor, why the rubber particles $500\text{ }\mu\text{m}+$ still appeared, in total 14 sieving runs were performed to obtain material for the further analyses. Only rubber particles, which were collected on sieves below $399\text{ }\mu\text{m}$, were stored.

Re-sieving of the collected particles under the optimized conditions

Similarly, at the optimized time of 60 min and the mass of 200 g, three sieving runs were performed for the sieved particles below $399\text{ }\mu\text{m}$, the results of which are represented in Fig. 25, showing the percentage fraction of the retained particles versus the mesh size of the sieves. The particles were re-sieved to confirm the size of the particle which was sieved before from their size below $300\text{ }\mu\text{m}$ particles were taken and re-sieved and found that still there were some particles left $500\text{ }\mu\text{m}+$ shown in the Table 2.

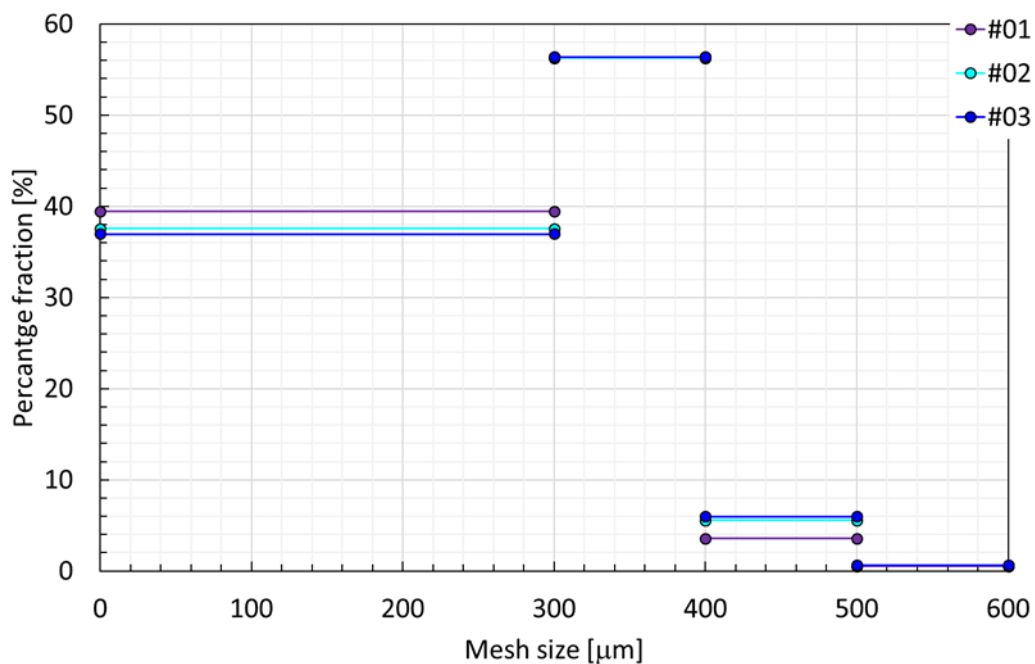


Fig. 25 Percentage fraction of rubber particles obtained from collected material ($0\text{--}299\text{ }\mu\text{m} + 300\text{--}399\text{ }\mu\text{m}$) under optimized conditions

Although, now the sieving was done with the collected particles below $400\text{ }\mu\text{m}$ under optimized conditions, yet, the results were not at all satisfactory, because a large percentage retention above $400\text{ }\mu\text{m}$ and $500\text{ }\mu\text{m}+$ appeared after sieving.

Table 2 Percentage fraction of re-sieved rubber particles at different runs

Mess size[μm]	Run#01 [%]	Run#02 [%]	Run#03 [%]
500+	0.55	0.60	0.65
400–499	3.59	5.58	5.98

As the next steps, rubber particles were treated in various ways.

Drying/re-sieving of the collected particles under the optimized conditions

For further sieving experiments, the collected particles (0–299 μm + 300–399 μm) were dried under conditions mentioned above. After drying, three runs at the optimized time and mass were performed on the collected samples. Fig. 26 reflects the obtained results where the percentage fraction of the retained particles versus the mesh size of the sieves were plotted.

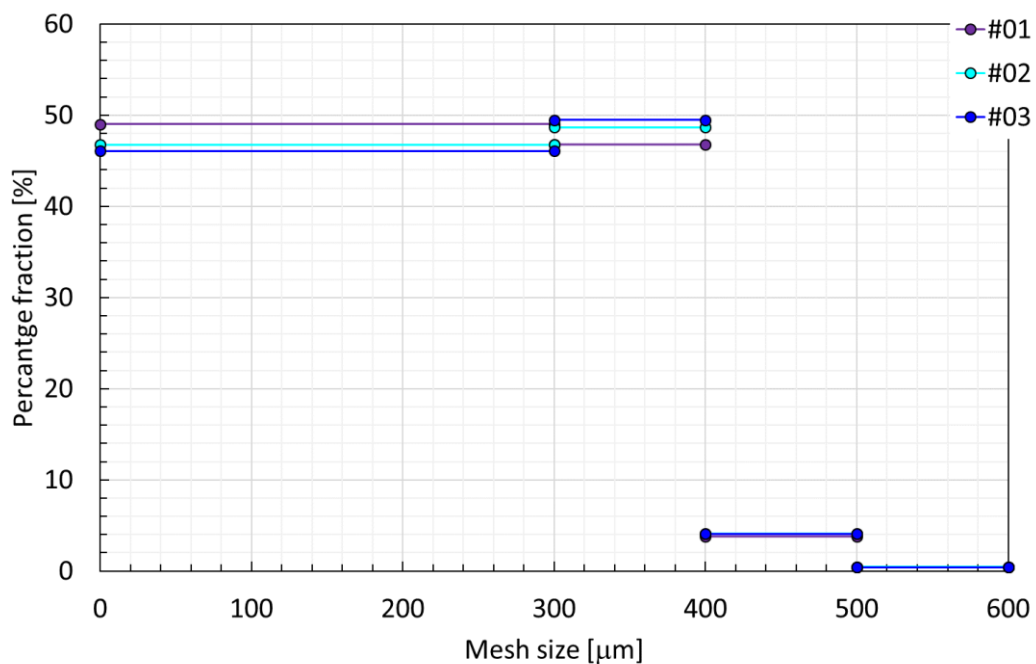


Fig. 26 Percentage fraction of dried rubber particles obtained from collected material under optimized conditions

The result showed lesser percentage 500 $\mu\text{m}+$ than that of the original particles. The data is shown in Table 3.

Table 3 Percentage fraction of dried rubber particles at different runs

Mess size [μm]	Run#01 [%]	Run#02 [%]	Run#03 [%]
500+	0.42	0.45	0.40
400–499	3.82	4.09	4.07

Acetone treatment/re-sieving of the collected particles under the optimized conditions

Another modification was done by treating the collected particles (0–299 μm + 300–399 μm) with acetone. Three runs at the optimized time and mass, were performed on the acetone-treated samples. Fig. 27 reflects the obtained results where the percentage fraction of the retained particles versus the mesh size of the sieves were plotted.

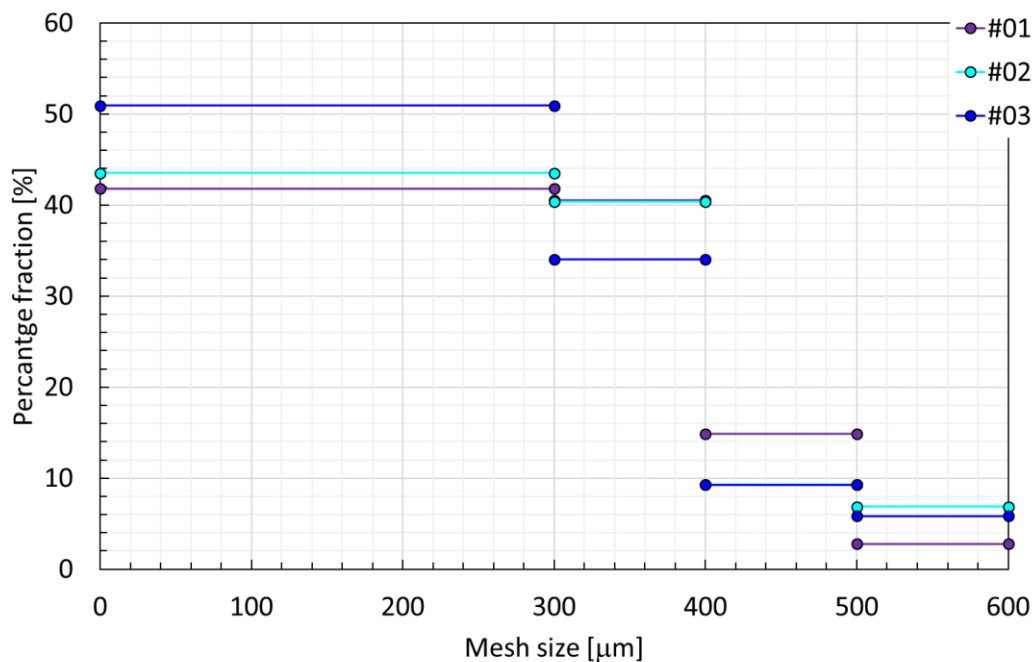


Fig. 27 Percentage fraction of rubber particles treated in acetone obtained from collected material under optimized conditions

It was hypothesized that the acetone did not get properly removed, causing the generation of agglomerates, resulting in the unwanted higher retention on the higher mesh dimensions shown in the Table 4.

Table 4 Percentage fraction of rubber particles treated in acetone at different runs

Mess size[μm]	Run#01 [%]	Run#02 [%]	Run#03 [%]
500+	2.78	6.86	5.82
400–499	14.89	9.26	9.26

Drying/re-sieving of the collected particles under the optimized conditions

To check the effect of smaller size particles on percentage fraction, collected particles in particles range 0–299 μm were taken, dried and re-sieved again with three runs at the optimized time and mass. Fig. 28 reflects the obtained results where the percentage fraction of the retained particles versus the mesh size of the sieves were plotted.

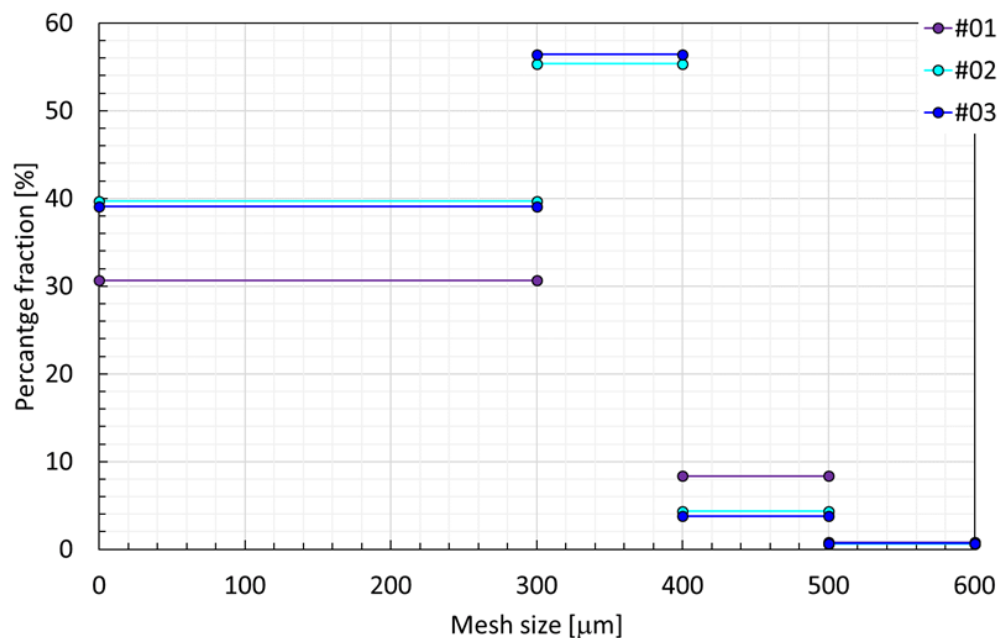


Fig. 28 Percentage fraction of rubber particles obtained from collected material treated in acetone under optimized conditions

Perhaps the result was shocking there were again some particles left above 400 μm and 500 μm sieve the data shown in the Table.5.

Table 5 Percentage fraction of dried rubber particles at different runs

Mess size[μm]	Run#01 [%]	Run#02 [%]	Run#03 [%]
500+	2.78	6.86	5.82
400–499	14.89	9.26	9.26
300–399	60.15	55.37	56.43

To understand the previous results and try to solve problems, which arose due to unwanted percentage retention 500 μm +, characterization techniques such as optical microscopy, TG analysis, and Fourier transform IR, were employed.

Optical microscopy

Optical microscopy was performed to understand the shape and size of rubber particles as one of potential reasons of the obtained results. Figs. 29–34.

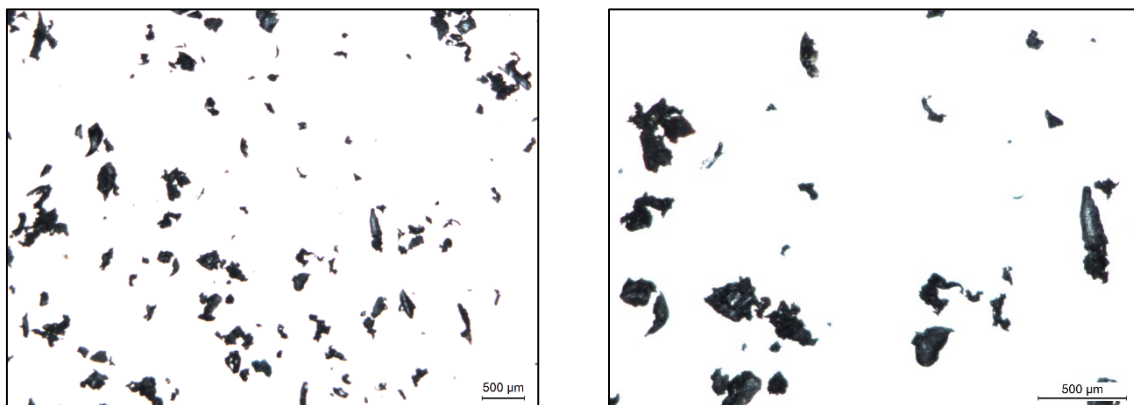


Fig. 29 Optical microscopy images of original rubber particles

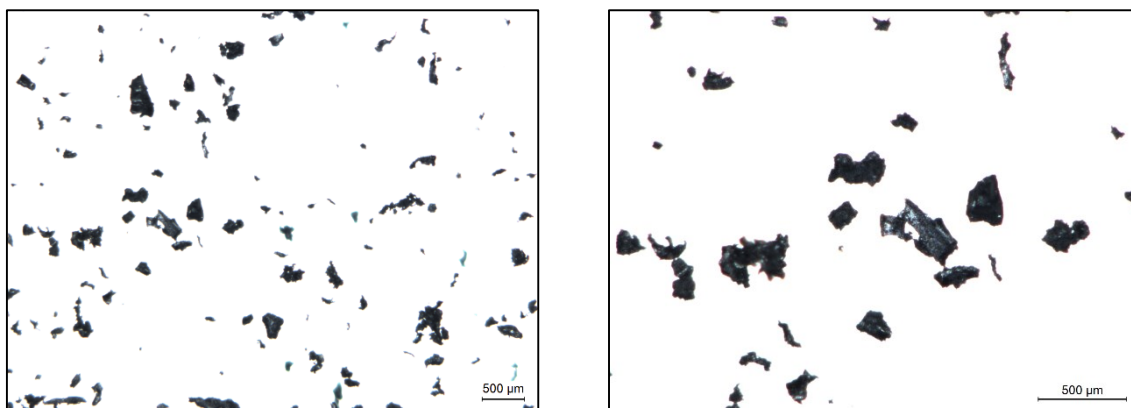


Fig. 30 Optical microscopy images of collected particles with the size of 0–299 μm

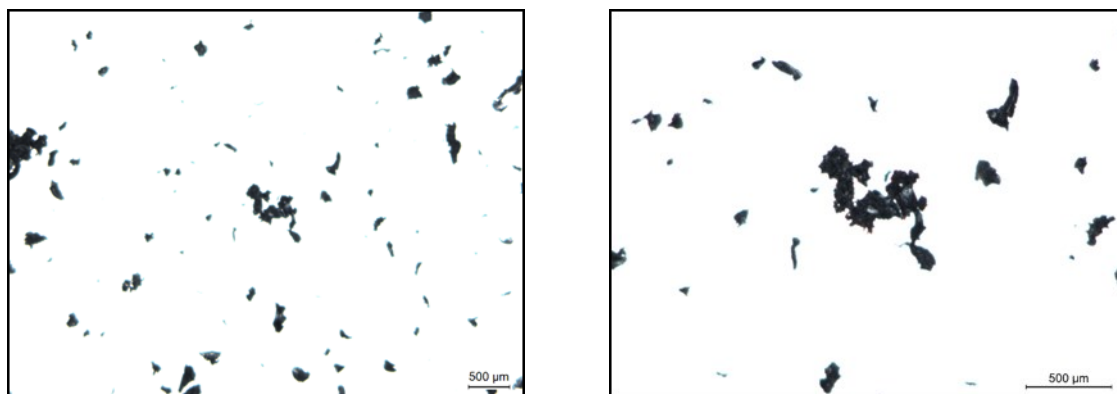


Fig. 31 Optical microscopy images of collected particles with the size of 300–399 μm

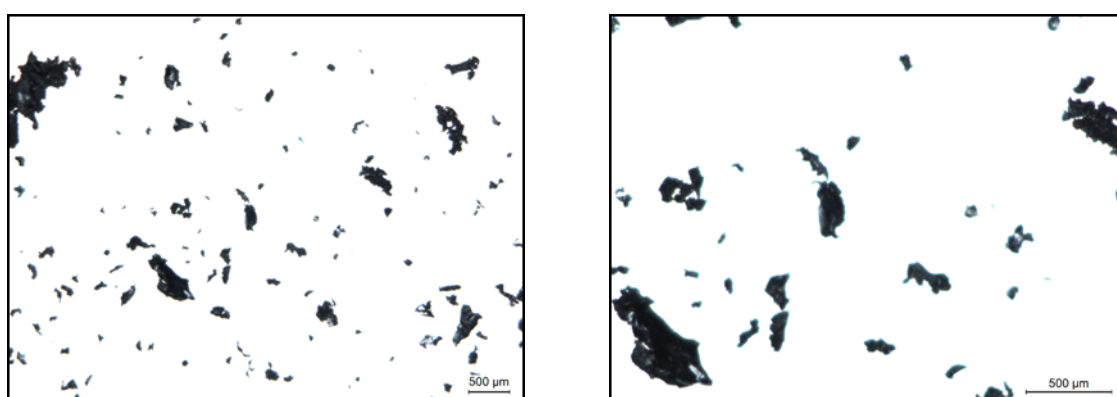


Fig. 32 Optical microscopy images of collected particles with the size of 400–499 μm

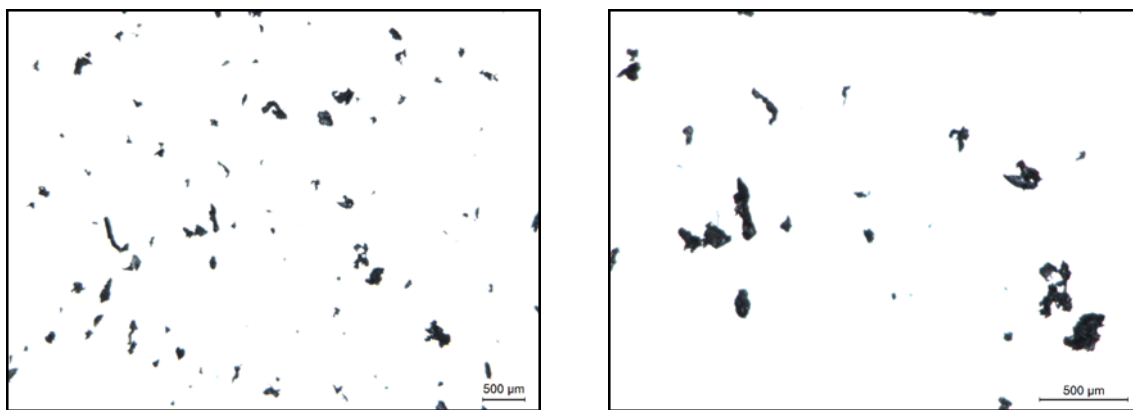


Fig. 33 Optical microscopy images of dried collected particles with the size of 300–399 μm

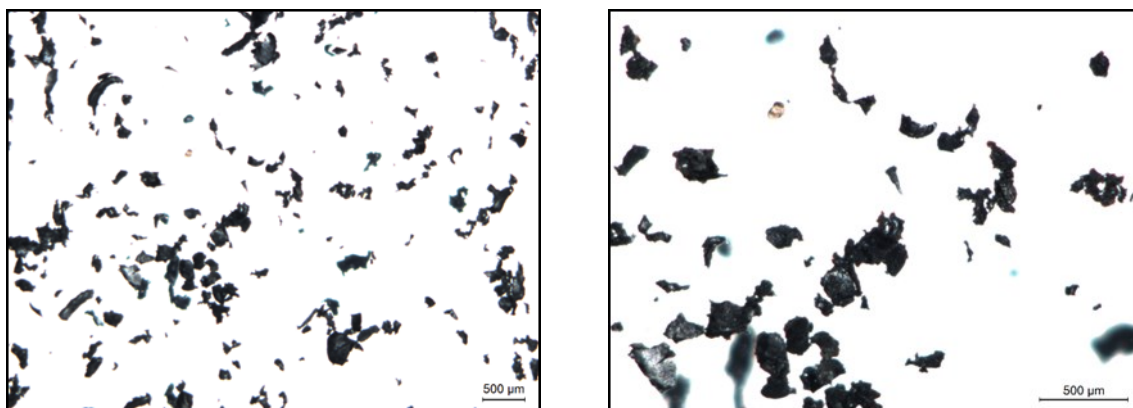


Fig. 34 Optical microscopy images of collected particles treated in acetone with the size of 300–399 μm

In optical microscopy, there was no observable difference before and after treatment as compared to all the results, and the size of the samples were similar. The only difference was in the structure. Before treatment, the shape of the particles got interlocked, and during sieving, the particles that were parallel to the vertical axis passed through the sieve easily, but the particles that were perpendicular to the sieve axis couldn't pass through the sieve. So, particle agglomeration could have been a potential reason for this.

Hence, acetone treatment was performed to overcome particle agglomeration. To check whether the particle agglomeration is possible or not, optical microscopy was performed. It was observed from the optical microscopy that the structure was not the same for all original rubber particles and treated ones at different sizes which is shown in the Figs. 30–34.

TG analysis

TG analysis was utilized to identify and quantify the wax component in analysed particles, which should be the focus of discussion on the present work. Apart of that, natural rubber, a mixture of styrene-butadiene rubber and polybutadiene rubber were also detected. Though the detection of other components such as carbon black, silica filler, and other residues such as zinc oxide could have been done, yet related to the present work, they were not important and lied outside the scope of the present discussion.

From the TG analysis, five regions were studied, namely, the degradation at a temperature of about 225 °C for the wax, at around 359 °C for natural rubber, at around 432 °C for the mixture of styrene-butadiene rubber and polybutadiene rubber, at around 663 °C for carbon black and one for residual mass as shown in Fig. 35.

It was found that for particle treated in acetone, the wax percent was significantly lower as compared to original particles (Table 6).

Table 6 Percentage concentration for individual components from TG analysis

Sample Designation	Wax	Natural rubber	Styrene-butadiene rubber + polybutadiene rubber	Carbon black	Residue
	T_{max} [°C]/ mass [%]	T_{max} [°C]/ mass [%]	T_{max} [°C]/ mass [%]	mass [%]	mass [%]
Original particles	225/4.4	359/34.6	432/26.5	20.9	13.6
Acetone treated particles	217/2.5	358/33.9	432/27.2	21.2	15.2

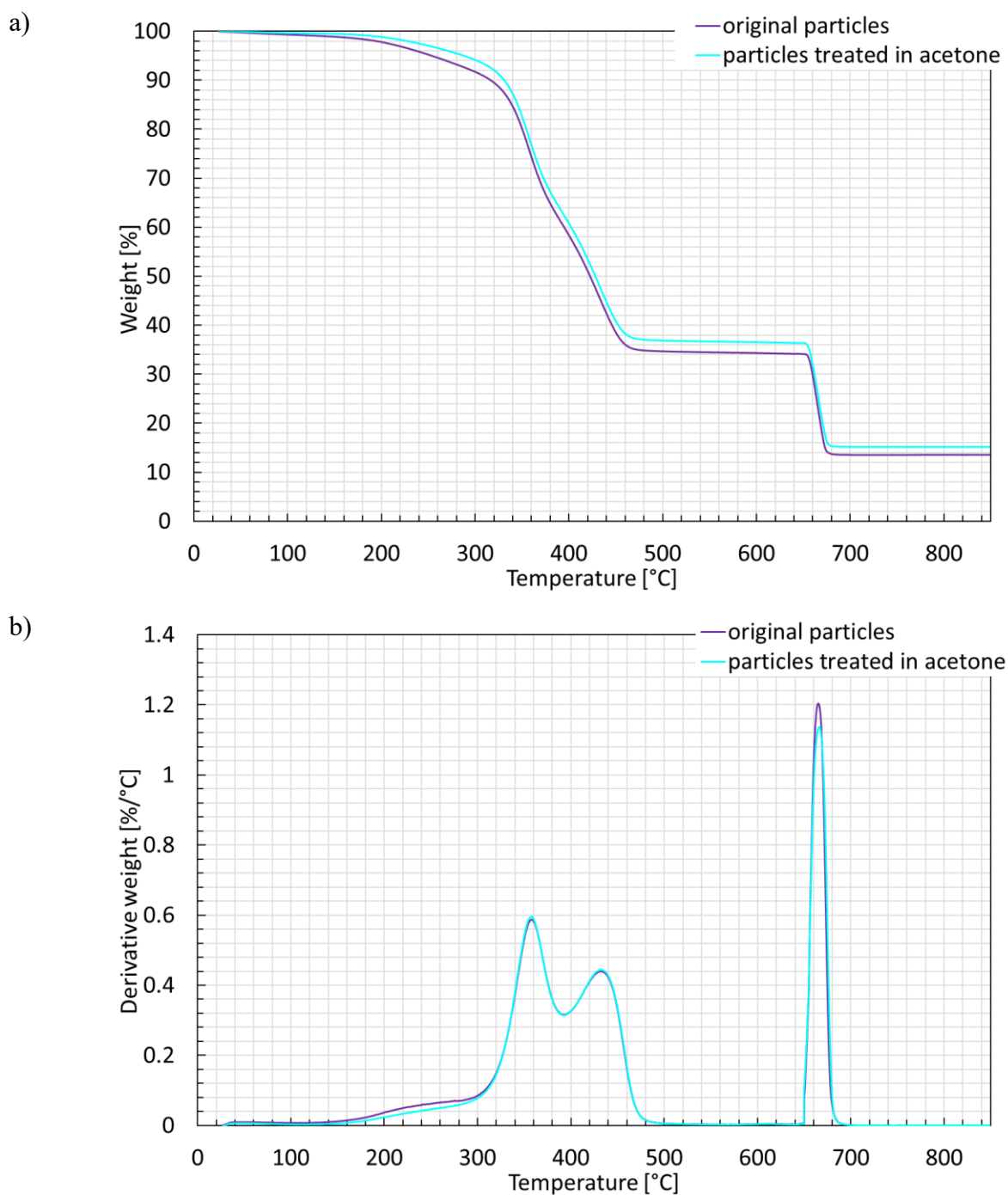


Fig. 35 Representative TG thermographs of a) weight loss, and b) its derivative versus temperature for original rubber particles and particles treated in acetone

The details of the calculations to convert mass percent to phr is as follows:

mass of natural rubber [%] + mass of (styrene-butadiene rubber [%] + polybutadiene rubber [%]) $\equiv$ 100 phr

Therefore, phr of any component $\equiv 100/((\text{mass of natural rubber [\%]} + \text{mass of (styrene-butadiene rubber [\%]} + \text{polybutadiene rubber [\%])}) \cdot \text{component [\%]}$

The percent mass as obtained from the TG analysis was converted to phr (Table 7).

Table 7 Concentration in phr for individual components from TG analysis

Sample Designation	Wax	Natural rubber	Styrene-butadiene rubber + polybutadiene rubber	Carbon black	Residue
	[phr]				
Original particles	7.2	56.7	43.3	34.2	22.3
Acetone treated particles	4.1	55.5	44.5	34.8	24.9

The TG analysis and its derivation successfully detected and quantified the wax, natural rubber, a mixture of styrene-butadiene rubber and polybutadiene rubber, carbon black, and residues (mostly silica) in the rubber particles. The observed degradation peaks gave strong proof of the presence and quantities of natural rubber and the mixture of styrene-butadiene rubber and polybutadiene rubber. The change in the concentration of the wax content by the acetone treatment was detected.

The relevance of this finding in detecting different amounts of wax on original particles and particles treated by acetone were further corroborated by the findings of FT-IR studies.

Infrared spectroscopy

IR spectroscopy data as obtained from the IR instrument may frequently contain background noise, baseline shifting, and other interferences that might mask the sample's true spectrum properties. Correct baseline fitting with subsequent baseline subtraction to get the

baseline subtracted spectrum is of paramount importance which may then be judiciously used for quantifying the characteristic IR absorbance peaks of rubbers. Baseline subtraction was obtained by using well-established baseline subtraction method reported in literature [66].

IR spectra obtained for the original rubber particles, dried rubber particles, and acetone treated one are shown in Fig. 36.

In all the obtained IR spectra, the presence of natural rubber indicated by the characteristic absorbance peak at 1375 cm^{-1} , presence of styrene-butadiene rubber reflected through the peak at 699 cm^{-1} and presence of the wax showing the peak at 719 cm^{-1} were detected.

Table 8 shows the relative concentration of wax with respect to rubber components presented in the particles.

Table 8 Relative concentration of the wax for different samples

Samples designation	Concentration of wax with respect to natural rubber normalized to the concentration of 1
Original particles	* 0.16 ± 0.06
Particles treated in acetone	0.14 ± 0.05
Dried particles	0.37 ± 0.06

* Arithmetic mean for three readings with standard deviation based on sample

Thus, the findings from IR spectroscopy as related to the amount of the wax present in the original particles, particles treated in acetone, and dried particles, corroborated with the results that were obtained from TG analysis, showing that the wax content on the surface of the rubber particles significantly increased upon drying the original particles.

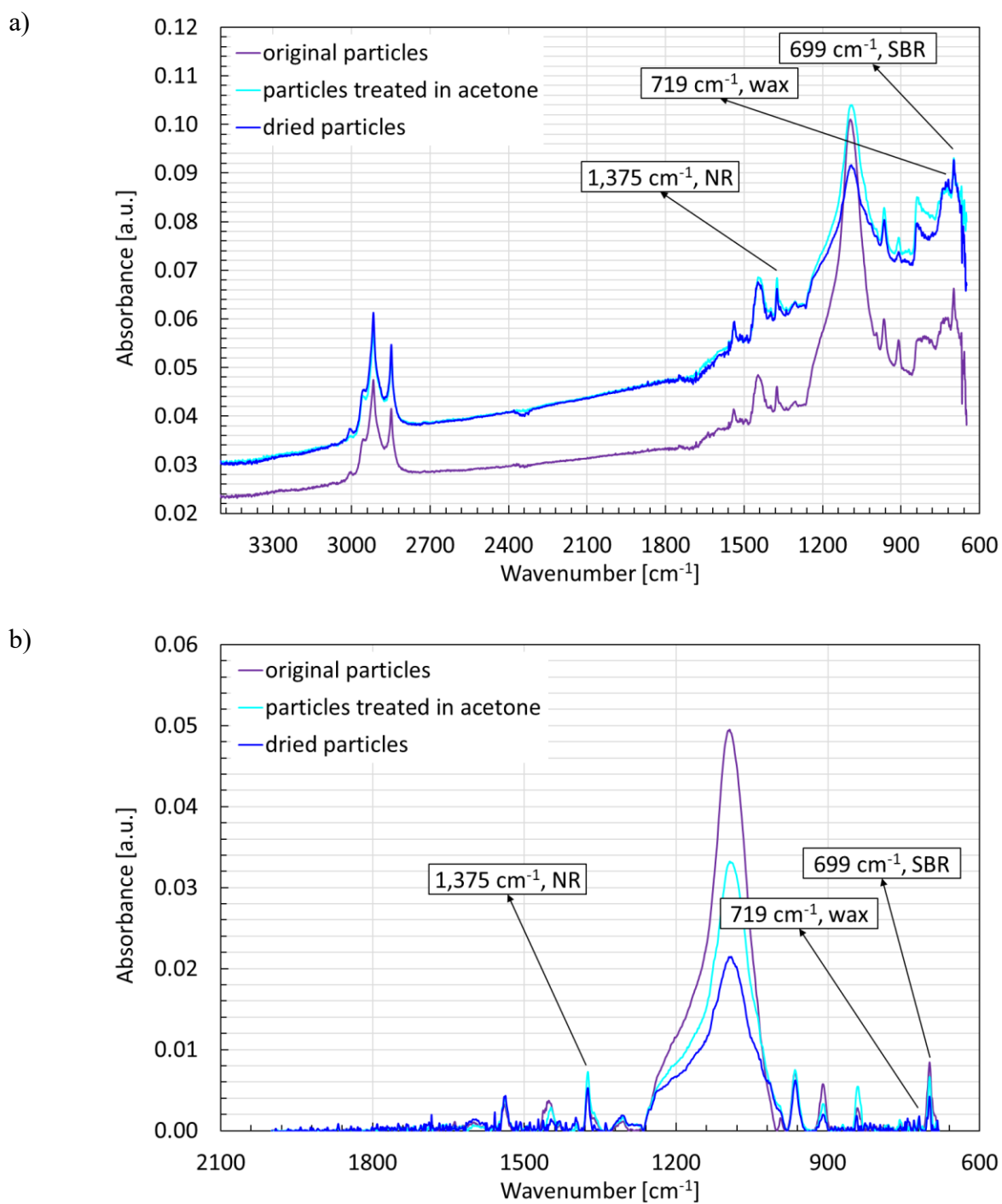


Fig. 36 Representative IR spectra of absorbance versus wavenumber: a) in original scale, and b) after baseline correction for original rubber particles, particles treated in acetone and dried particles

CONCLUSIONS

The project aimed at obtaining the maximum mass percent of the provided rubber particles below 500 μm , where supplied particles were claimed to have size below 500 μm , but observable retention above this dimension was there.

When the sieving was done with the original rubber particles it was found that a significant percentage were found to be retained 500 $\mu\text{m}+$. When re-sieving below 400 μm was done, it was again found that some particles were retained 500 $\mu\text{m}+$, and this was theoretically to be due to the formation of agglomerates and interlocking and also due to the different surface orientations. These different orientations allowed/disallowed a particular particle to pass through the sieve, and were detected using optical microscopy.

The project aimed to find a suitable treatment or the suitable number of treatments, physical and chemical, to reduce this unwanted percentage retention to a minimum. Two treatments were done which were as follows:

- The particles were dried before re-sieving,
- The particles were treated in acetone and dried and then re-sieved.

As it has been evidenced in Result and Discussion, it is reiterated that the amount of the wax substantially increased on the surface of the particles upon drying, which reduced the percentage retention 500 $\mu\text{m}+$.

This reduction partially shows the way how to solve the problem. However, the main impediment is to overcome difficulty arising from irregular shape and size of rubber particles due to grinding process.

Future work aims at different materials under different particle reduction conditions to optimise the factor affecting the desired requirements.

REFERENCES

- [1] Abbas, S., Fatima, A., Kazmi, S. M. S., Munir, M. J., Ali, S., & Rizvi, M. A. Effect of particle sizes and dosages of rubber waste on the mechanical properties of rubberized concrete composite. *Applied Sciences* **2022**, 12(17), 8460.
- [2] Roberts, A. D. *Natural rubber science and technology*; Oxford University Press, **1988**.
- [3] <https://www.statista.com/statistics/275399/world-consumption-of-natural-and-synthetic-caoutchouc/>
- [4] Isayev, A. I. Recycling of rubbers. in ‘Science and technology of rubber’(eds.: Mark JE, Erman B., Eirich F. R.) Elsevier. *New York* **2005**, 3, 663–701.
- [5] ISO 14040. *Environmental management—life cycle assessment—principles and framework* **2006**, 235–248.
- [6] Zedler, Ł.; Przybysz, M.; Klein, M.; Saeb, M. R.; Formela, K. Processing, physico-mechanical and thermal properties of reclaimed GTR and NBR/reclaimed GTR blends as function of various additives. *Polymer Degradation and Stability* **2017**, 143, 186–195.
- [7] Wang, Z.; Yuan, L.; Tang, C. Sustainable elastomers from renewable biomass. *Accounts of chemical research* **2017**, 50 (7), 1762–1773.
- [8] Lewandowski, L. H. Polymer modification of paving asphalt binders. *Rubber Chemistry and Technology* **1994**, 67 (3), 447–480.
- [9] <https://www.rainforest-alliance.org/species/rubber-tree/>
- [10] Wang, S.; Yan, Y.; Gao, X.; Zhang, H.; Cui, Y.; He, Q.; Wang, Y.; Wang, X. Emission characteristics and health risks of volatile organic compounds (VOCs) measured in a typical recycled rubber plant in China. *International Journal of Environmental Research and Public Health* **2022**, 19, 8753. DOI: 10.3390/ijerph19148753.
- [11] Elias, H.-G. *An introduction to plastics*; John Wiley & Sons, **2003**.
- [12] Chakraborty, S.; Bandyopadhyay, S.; Ameta, R.; Mukhopadhyay, R.; Deuri, A. S. Application of FTIR in characterization of acrylonitrile-butadiene rubber (nitrile rubber). *Polymer Testing* **2007**, 26 (1), 38–41.

- [13] Kresge, E.; Wang, H. C. Butyl rubber. *Kirk-Othmer Encyclopedia of Chemical Technology* **2000**.
- [14] Hasegawa, N.; Okamoto, H.; Usuki, A. Preparation and properties of ethylene propylene rubber (EPR)–clay nanocomposites based on maleic anhydride-modified EPR and organophilic clay. *Journal of applied polymer science* **2004**, 93 (2), 758–764.
- [15] Anbazhagan, P.; Manohar, D. R.; Rohit, D. Influence of size of granulated rubber and tyre chips on the shear strength characteristics of sand–rubber mix. *Geomechanics and Geoengineering* **2017**, 12 (4), 266–278.
- [16] Noriman, N. Z.; Ismail, H.; Rashid, A. A. Characterization of styrene butadiene rubber/recycled acrylonitrile-butadiene rubber (SBR/NBRr) blends: The effects of epoxidized natural rubber (ENR-50) as a compatibilizer. *Polymer Testing* **2010**, 29 (2), 200–208.
- [17] Tamanaha, C. R. *Feasibility study of biomembrane reconstitution and transduction mechanisms on inorganic microporous ceramic filters*; Worcester Polytechnic Institute, **1997**.
- [18] Bandyopadhyay, S.; Agrawal, S. L.; Ameta, R.; Dasgupta, S.; Mukhopadhyay, R.; Deuri, A. S.; Ameta, S. C.; Ameta, R. An overview of rubber recycling. *Progress in Rubber Plastics and Recycling Technology* **2008**, 24 (2), 73–112.
- [19] Fazli, A.; Rodrigue, D. Waste rubber recycling: A review on the evolution and properties of thermoplastic elastomers. *Materials* **2020**, 13 (3), 782.
- [20] Formela, K.; Hejna, A.; Zedler, L.; Colom Fajula, X.; Cañavate Ávila, F. J. Microwave treatment in waste rubber recycling–recent advances and limitations. *Express polymer letters* **2019**, 13 (6), 565–588.
- [21] Kern, W. J.; Futamura, S. Effect of tread polymer structure on tyre performance. *Polymer* **1988**, 29 (10), 1801–1806.
- [22] Loadman, J. *Tears of the tree: the story of rubber-a modern marvel*; OUP Oxford, **2005**.
- [23] Kim, J. K.; Saha, P.; Thomas, S.; Haponiuk, J. T.; Aswathi, M. K. Rubber recycling: challenges and developments; *Royal Society of Chemistry*, **2018**.

- [24] Hong, Z.; Bo, L.; Guangsu, H.; Jia, H. A novel composite sound absorber with recycled rubber particles. *Journal of Sound and Vibration* **2007**, *304* (1-2), 400–406.
- [25] Rashad, A. M. A comprehensive overview about recycling rubber as fine aggregate replacement in traditional cementitious materials. *International Journal of Sustainable Built Environment* **2016**, *5* (1), 46–82.
- [26] Sienkiewicz, M.; Kucinska-Lipka, J.; Janik, H.; Balas, A. Progress in used tyres management in the European Union: A review. *Waste Management* **2012**, *32* (10), 1742–1751.
- [27] Archibong, F. N.; Sanusi, O. M.; Médéric, P.; Hocine, N. A. An overview on the recycling of waste ground tyre rubbers in thermoplastic matrices: Effect of added fillers. *Resources, Conservation and Recycling* **2021**, *175*, 105894.
- [28] Xiao, Z.; Pramanik, A.; Basak, A. K.; Prakash, C.; Shankar, S. Material recovery and recycling of waste tyres-A review. *Cleaner Materials* **2022**, 100115.
- [29] Moreno, T.; Balasch, A.; Bartrolí, R.; Eljarrat, E. A new look at rubber recycling and recreational surfaces: The inorganic and OPE chemistry of vulcanised elastomers used in playgrounds and sports facilities. *Science of The Total Environment* **2023**, 161648.
- [30] Valentini, F.; Pegoretti, A. End-of-life options of tyres. A review. *Advanced Industrial and Engineering Polymer Research* **2022**.
- [31] Tauberova, R.; Marticek, M.; Knapcikova, L. Selected innovative approaches in the waste tyres management. *Acta Logistica* **2022**, *9* (4), 411–415.
- [32] Zedler, Ł.; Wang, S.; Formela, K. Ground tire rubber functionalization as a promising approach for the production of sustainable adsorbents of environmental pollutants. *Science of the Total Environment* **2022**, 155636.
- [33] Phiri, M. M.; Phiri, M. J.; Formela, K.; Wang, S.; Hlangothi, S. P. Grafting and reactive extrusion technologies for compatibilization of ground tyre rubber composites: Compounding, properties, and applications. *Journal of Cleaner Production* **2022**, 133084.

- [34] Formela, K. Analysis of volatile organic compounds emission in the rubber recycling products quality assessment. *Advanced Industrial and Engineering Polymer Research* **2022**.
- [35] Saeb, M. R.; Wiśniewska, P.; Susik, A.; Zedler, Ł.; Vahabi, H.; Colom, X.; Cañavate, J.; Tercjak, A.; Formela, K. GTR/Thermoplastics blends: how do interfacial interactions govern processing and physico-mechanical properties? *Materials* **2022**, *15* (3), 841.
- [36] Zitzumbo, R.; Becerra, M. B.; Padilla-Rizo, B.; Estrada-Monje, A.; Alonso-Romero, S. Mechanical and performance properties of re-vulcanized blends of GTR/NR. *Progress in Rubber, Plastics and Recycling Technology* **2022**, 14777606221118637.
- [37] Kiss, L.; Simon, D. Á.; Petrény, R.; Kocsis, D.; Bárány, T.; Mészáros, L. Ground tire rubber filled low-density polyethylene: The effect of particle size. *Advanced Industrial and Engineering Polymer Research* **2022**, *5* (1), 12–17.
- [38] Fazli, A.; Rodrigue, D. Recycling waste tires into ground tire rubber (GTR)/rubber compounds: A review. *Journal of Composites Science* **2020**, *4* (3), 103.
- [39] Behnood, A.; Olek, J. Rheological properties of asphalt binders modified with styrene-butadiene-styrene (SBS), ground tire rubber (GTR), or polyphosphoric acid (PPA). *Construction and Building Materials* **2017**, *151*, 464–478.
- [40] Simon, D. Á.; Piritzyi, D.; Tamás-Bényei, P.; Bárány, T. Microwave devulcanization of ground tire rubber and applicability in SBR compounds. *Journal of Applied Polymer Science* **2020**, *137* (6), 48351.
- [41] Karger-Kocsis, J.; Mészáros, L.; Bárány, T. Ground tyre rubber (GTR) in thermoplastics, thermosets, and rubbers. *Journal of Materials Science* **2013**, *48*, 1–38.
- [42] Wang, Z.; Zeng, D. Preparation of devulcanized ground tire rubber with supercritical carbon dioxide jet pulverization. *Materials Letters* **2021**, *282*, 128878.
- [43] Nadal Gisbert, A.; Crespo Amorós, J. E.; López Martínez, J.; Garcia, A. M. Study of thermal degradation kinetics of elastomeric powder (ground tire rubber). *Polymer-Plastics Technology and Engineering* **2007**, *47* (1), 36–39.

- [44] Karabork, F.; Pehlivan, E.; Akdemir, A. Characterization of styrene butadiene rubber and microwave devulcanized ground tire rubber composites. *Journal of Polymer Engineering* **2014**, *34* (6), 543–554.
- [45] Sonnier, R.; Leroy, E.; Clerc, L.; Bergeret, A.; Lopez-Cuesta, J. M. Polyethylene/ground tyre rubber blends: Influence of particle morphology and oxidation on mechanical properties. *Polymer Testing* **2007**, *26* (2), 274–281.
- [46] Chiu, C.-T.; Lu, L.-C. A laboratory study on stone matrix asphalt using ground tire rubber. *Construction and Building Materials* **2007**, *21* (5), 1027–1033.
- [47] Zhang, X.; Lu, C.; Liang, M. Properties of natural rubber vulcanizates containing mechanochemically devulcanized ground tire rubber. *Journal of Polymer Research* **2009**, *16*, 411–419.
- [48] Zefeng, W.; Yong, K.; Zhao, W.; Yi, C. Recycling waste tire rubber by water jet pulverization: Powder characteristics and reinforcing performance in natural rubber composites. *Journal of Polymer Engineering* **2018**, *38* (1), 51–62.
- [49] Zedler, Ł.; Kowalkowska-Zedler, D.; Colom, X.; Cañavate, J.; Saeb, M. R.; Formela, K. Reactive sintering of ground tire rubber (GTR) modified by a trans-polyoctenamer rubber and curing additives. *Polymers* **2020**, *12* (12), 3018.
- [50] Xiao, Q.; Cao, C.; Xiao, L.; Bai, L.; Cheng, H.; Lei, D.; Sun, X.; Zeng, L.; Huang, B.; Qian, Q. Selective decomposition of waste rubber from the shoe industry by the combination of thermal process and mechanical grinding. *Polymers* **2022**, *14* (5), 1057.
- [51] Lapkovskis, V.; Mironovs, V.; Kasperovich, A.; Myadelets, V.; Goljandin, D. Crumb rubber as a secondary raw material from waste rubber: A short review of end-of-life mechanical processing methods. *Recycling* **2020**, *5* (4), 32.
- [52] Lee, S. H.; Hwang, S. H.; Kontopoulou, M.; Sridhar, V.; Zhang, Z. X.; Xu, D.; Kim, J. K. The effect of physical treatments of waste rubber powder on the mechanical properties of the revulcanizate. *Journal of Applied Polymer Science* **2009**, *112* (5), 3048–3056.
- [53] Fisher, H. L. Vulcanization of rubber. *Industrial & Engineering Chemistry* **1939**, *31* (11), 1381–1389.

- [54] Craig, D. The vulcanization of rubber with sulfur. *Rubber Chemistry and Technology* **1957**, 30 (5), 1291–1346.
- [55] Stephens, H. L. The compounding and vulcanization of rubber. *Rubber Technology* **1999**, 20–58.
- [56] Shi, J.; Jiang, K.; Ren, D.; Zou, H.; Wang, Y.; Lv, X.; Zhang, L. Structure and performance of reclaimed rubber obtained by different methods. *Journal of Applied Polymer Science* **2013**, 129 (3), 999–1007.
- [57] Rubber, S. N. Natural rubber and reclaimed rubber composites—A systematic review. *Polymer* **2016**, 2 (7).
- [58] Le Beau, D. S. Science and technology of reclaimed rubber. *Rubber Chemistry and Technology* **1967**, 40 (1), 217–237.
- [59] Rattanasom, N.; Poonsuk, A.; Makmoon, T. Effect of curing system on the mechanical properties and heat aging resistance of natural rubber/tire tread reclaimed rubber blends. *Polymer testing* **2005**, 24 (6), 728–732.
- [60] Jia, M.; Peinemann, K.-V.; Behling, R.-D. Molecular sieving effect of the zeolite-filled silicone rubber membranes in gas permeation. *Journal of Membrane Science* **1991**, 57 (2-3), 289–292.
- [61] Chen, C.; Liu, W.; Jiang, X.; Wu, J. Effects of rubber-based agroforestry systems on soil aggregation and associated soil organic carbon: Implications for land use. *Geoderma* **2017**, 299, 13–24.
- [62] Indraratna, B.; Qi, Y.; Heitor, A. Evaluating the properties of mixtures of steel furnace slag, coal wash, and rubber crumbs used as subballast. *Journal of Materials in Civil Engineering* **2018**, 30 (1), 04017251.
- [63] <https://www.cas.miamioh.edu/mbi-ws/microscopes/compoundscope.html>
- [64] <https://www.linseis.com/en/methods/thermogravimetric-analysis/>
- [65] <https://specac.com/theory-articles/introduction-to-atr-ftir-spectroscopy-part-1-the-basics/>
- [66] Datta, S.; Antos, J.; Stoczek, R. Smart numerical method for calculation of simple general infrared parameter identifying binary rubber blends. *Polymer Testing* **2017**, 57, 192–202.

LIST OF FIGURES

Fig. 1 Different particle size waste rubber [1]	10
Fig. 2 Waste tyre recycling production [2]	11
Fig. 3 Consumption of different types of rubbers in metric tons (= 1,000 kg) [3]	12
Fig. 4 Dumping of tyre waste [5]	13
Fig. 5 Natural rubber latex [12]	16
Fig. 6 Typical process flowchart of waste rubber tyre recycling [17]	18
Fig. 7 Burning of discarded tyres	22
Fig. 8 Sieve Analyser	30
Fig. 9 Ground rubber sample	33
Fig. 10 Different sieves with corresponding fractions: a) 500 μm , b) 400 μm , c) 300 μm , and d) dish	36
Fig. 11 Sample prepared for drying	37
Fig. 12 Oven condition for drying	38
Fig. 13 Extracted acetone sample	39
Fig. 14 Extracted sample for drying	39
Fig. 15 Drying conditions after acetone treatment	40
Fig. 16 Optical microscope: a) Principle [75] and b) LeicaDVM-2500	41
Fig. 17 TG analysis: a) Principle [76], b) TG analyser Q500-1788, and c) detail of sample holder and analysed sample	43
Fig. 18 IR spectroscopy: a) Principle [77], and b) detail of ATR cell with germanium crystal	44
Fig. 19 Percentage fraction of original rubber particles for various times of sieve analysis for run 1	45
Fig. 20 Percentage fraction of original rubber particles for various times of sieve analysis run 2	46
Fig. 21 Fraction of 500 μm^+ collected at different times of sieve analysis	47
Fig. 22 Percentage fraction of original rubber particles for amount of sieved material for run 1	48
Fig. 23 Percentage fraction of original rubber particles for amount of sieved material for run 2	48
Fig. 24 Percentage fraction of original rubber particles obtained under optimized conditions	49

Fig. 25 Percentage fraction of rubber particles obtained from collected material (0–299 μm + 300–399 μm) under optimized conditions.....	50
Fig. 26 Percentage fraction of dried rubber particles obtained from collected material under optimized conditions.....	51
Fig. 27 Percentage fraction of rubber particles treated in acetone obtained from collected material under optimized conditions.....	52
Fig. 28 Percentage fraction of rubber particles obtained from collected material treated in acetone under optimized conditions.....	53
Fig. 29 Optical microscopy images of original rubber particles.....	54
Fig. 30 Optical microscopy images of collected particles with the size of 0–299 μm	55
Fig. 31 Optical microscopy images of collected particles with the size of 300–399 μm	55
Fig. 32 Optical microscopy images of collected particles with the size of 400–499 μm	55
Fig. 33 Optical microscopy images of dried collected particles with the size of 300–399 μm	56
Fig. 34 Optical microscopy images of collected particles treated in acetone with the size of 300–399 μm	56
Fig. 35 Representative TG thermographs of a) weight loss, and b) its derivative versus temperature for original rubber particles and particles treated in acetone.....	58
Fig. 36 Representative IR spectra of absorbance versus wavenumber: a) in original scale, and b) after baseline correction for original rubber particles, particles treated in acetone and dried particles.....	61

LIST OF TABLES

Table 1 Percentage fraction of rubber particles at different runs	49
Table 2 Percentage fraction of re-sieved rubber particles at different runs	51
Table 3 Percentage fraction of dried rubber particles at different runs	52
Table 4 Percentage fraction of rubber particles treated in acetone at different runs	53
Table 5 Percentage fraction of dried rubber particles at different runs	54
Table 6 Percentage concentration for individual components from TG analysis	57
Table 7 Concentration in phr for individual components from TG analysis	59
Table 8 Relative concentration of the wax for different samples	60

LIST OF SYMBOLS/ABBREVIATIONS

EOL	End of life
ETRMA	European Tyre and Rubber Manufacture's Association
scCO ₂	Super critical carbon dioxide
GTR	Ground tyre rubber
TG	Termogravimetric
ATR	Attenuation total reflection
IR	Infrared
ZnSe	Zinc selenide