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Innovative Method of Devulcanizing Waste Rubber from the Automotive Industry

H A Deepa [a*], Chandini N V [a], Himanshu Mishra [a], Priyanka Sahu [a] and R Neelesh [a]

[a] *Department of Chemical Engineering, Dayananda Sagar College of Engineering, Bengaluru-560111, Karnataka, India.*

Authors' contributions

This work was carried out in collaboration among all authors. Author RN introduced the concept of the manuscript. Authors CNV, HM, PS and RN did the methodology, visualization and formal analysis of the study. Author HAD supervised the manuscript. All authors wrote the original draft and reviewed the manuscript. Author PS edited the manuscript. All authors have read and approved the final version of the manuscript.

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ABSTRACT

Chemical devulcanization of waste rubber from the automotive industry was carried using benzoyl peroxide as devulcanizing agent and, o-xylene and toluene, as solvents. The effect of time on the process of devulcanizing was investigated. The extent of sulphur removal from the reclaimed rubber was studied by using EDAX technique. Thus, this devulcanized rubber can be blended with virgin rubber and on revulcanizing the mixture, it becomes possible to reuse waste rubber and give it a new life.

**Corresponding author: E-mail: deepaha@gmail.com;*

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Keywords: Devulcanization; waste rubber; o-xylene; toluene; benzoyl peroxide; sulphur removal; EDAX analysis.

1. INTRODUCTION

Approximately 70% of the world's rubber production is dedicated to tire manufacturing. The annual global discard of waste tires stands at around 800 million (equivalent to approximately 10 million tons), with 60% of this comprising natural and synthetic rubber, totalling approximately 6 million tons of tire scraps per year [1,2]. Despite various methods for handling used tires, the predominant practice involves landfill deposition, creating stockpiles that pose fire risks and foster environments conducive to rodents, mosquitoes, and other pests, thereby contributing to health and environmental concerns [3-5]. The European Commission banned the landfilling of end-of-life tires in 1999 [6], prompting extensive research in many countries on recycling and reutilization strategies.

Waste rubber finds diverse applications, including sports and playing surfaces, floor and walkway tiles, concrete, thermal and acoustic insulation, footwear, road and rail equipment, and activated carbon production [7-9]. However, the most effective approach to managing rubber waste is through recycling, encompassing various methods converting rubber waste into new commodities [7,10-15]. Literature suggests that rubber recycling is more energy-efficient compared to alternative methods such as burning [16].

The inherent complexity of rubber, characterized by a three-dimensional network structure, the robust bonds formed during vulcanization, and the diverse composition with additives, presents a formidable technological challenge in recycling. Tires, being composites, consist of various synthetic and natural rubbers, particulate fillers (silica, carbon black), chemical additives (sulphur, oils), and textile and/or metal reinforcements [17-20].

In the pursuit of recycling and repurposing vulcanized rubbers, it becomes paramount to identify a secure method for their devulcanization, involving the cleaving of their crosslink bonds. Rubber devulcanization is a transformative process wherein vulcanized waste rubber is converted into a state conducive to re-vulcanization after appropriate mixing and processing [21-24]. Specifically, devulcanization can be precisely characterized as a process that entirely or partially breaks poly-, di-, and mono-sulfidic bonds formed during vulcanization. This definition underscores the selective disruption of sulfidic bonds while preserving the polymer's main chain integrity, avoiding significant scissions or degradation.

To facilitate this process, sulphur-free toluene was selected as the solvent in our study, aiming to optimize the efficiency and environmental sustainability of the devulcanization process. This strategic combination of benzoyl peroxide as the devulcanizing agent and sulphur-free toluene as the solvent underscores our commitment to developing a greener and more effective solution for recycling waste tire rubber.

2. MATERIALS AND METHODS

2.1 Materials and Chemicals Used

Reclaimed rubber sheet was obtained from SM Enterprises, Bengaluru, India. The chemicals procured for devulcanization were sulphur-free o-Xylene (analytical grade), sulphur-free Toluene (analytical grade) and Benzoyl Peroxide (analytical grade).

2.2 Preparation of Rubber Samples

Reclaimed rubber sheets underwent precise cutting into uniform dimensions of 1cm x 1cm x 0.2cm using a utility knife, ensuring uniformity in the samples. The solvents opted for immersion of the rubber samples were sulphur-free o-Xylene, sulphur-free Toluene. 1 gram of the rubber pieces was immersed into the 20 ml of solvent, ensuring the rubber pieces were thoroughly immersed. Four samples were prepared, with two containing o-xylene as solvent and the other two containing toluene. Each solvent included 0.1 gram of benzoyl peroxide (BPO) to facilitate the breakdown of sulphur cross-links.

2.3 Devulcanization

Devulcanization refers to the process of breaking down or reversing the vulcanization of rubber, which involves the breaking of sulfur cross-links within the rubber polymer chains. This process incorporates heating the waste rubber samples at a certain temperature in the presence of devulcanizing agent. The prepared samples were subjected to controlled heating in a hot air oven set at 80℃. Two sets of samples underwent devulcanization for varying durations, one set for 1 hour and the other for 1.5 hours, allowing for comparative analysis. Upon completion of the devulcanization process, the treated rubber samples were carefully retrieved from the oven. Further processing involved separating the rubber from the solvent and subsequent drying in the oven to eliminate residual solvent, thus priming the samples for subsequent analysis.

(a) (b)

Fig. 1. Addition of solvent (a) o-Xylene & (b) Toluene and BPO to the reclaimed rubber

(a) (b)

Fig. 2. Samples in solvent (a) o-Xylene & (b) Toluene after treating for 1 hour at 80^0C

(a) (b)

Fig. 3. Devulcanized rubber obtained using (a) o-Xylene & (b) Toluene after drying for 10 mins at 80^0C

3. RESULTS AND DISCUSSION

To evaluate the effectiveness of sulphur removal in devulcanization process, the rubber samples before and after devulcanization were subjected to characterization using the EDAX technique, a powerful analytical tool that provides valuable insights into the elemental composition of materials.

The EDAX analysis conducted on the reclaimed rubber sample revealed crucial insights into its composition.

Table 1. Elemental composition by weight of untreated reclaimed rubber

Element	Weight %
C K	61.76
S K	2.73

From Fig. 4, the results indicated a substantial presence of carbon, accounting for 61.76% of the sample's weight, signifying the predominance of carbon-based compounds within the material. Additionally, the analysis identified a notable proportion of sulphur, constituting 2.73% of the sample's weight. These findings suggest the potential incorporation of both organic and inorganic compounds in the reclaimed rubber, highlighting its complex chemical makeup.

Table 2(a). Elemental composition by weight of sample treated with o-Xylene & BPO for 1 hour

Element	Weight %
C K	53.34
S K	1.78

Table 2(b). Elemental composition by weight of sample treated with Toluene & BPO for 1 hour

Element	Weight %
C K	57.59
S K	1.82

Following 1 hour treatment, the composition of the reclaimed rubber sample underwent significant changes compared to the initial analysis. Fig. 5(a) shows that in sample treated for 1 hour with o-xylene as solvent, the sulphur content decreased to 1.78% by weight, reflecting a reduction of 34.79% from the initial sulphur content. Additionally, the carbon content decreased to 53.34% by weight.

The analysis of the reclaimed rubber sample treated for 1 hour using toluene as solvent from Fig. 5(b) demonstrated a decrease in sulphur content to 1.82% by weight, indicating a reduction of 33.33% from the initial sulphur content. Additionally, carbon content decreased to 57.59% by weight, compared to the initial composition.

Table 3(a). Elemental composition by weight of sample treated with o-Xylene & BPO for 1.5 hours

Element	Weight %
C K	53.35
S K	1.56

Table 3(b). Elemental composition by weight of sample treated with Toluene & BPO for 1.5 hours

Element	Weight %
C K	48.19
S K	1.69

The analysis of the reclaimed rubber sample treated for 1.5 hours with o-xylene as solvent from Fig. 6(a) revealed that the sulphur content further decreased to 1.56% by weight, indicating a total reduction of 42.85% from the initial sulphur content. Also, a marginal change in carbon content, with 53.35% by weight was observed, compared to the previous result.

The reclaimed rubber sample treated for 1.5 hours using toluene as solvent upon analysis as shown in Fig. 6(b) revealed a further decrease in the sulphur content to 1.69% by weight, indicating a reduction of 38.09% from the initial sulphur content. Additionally, a decrease in carbon content to 48.19% by weight was observed, compared to previous results.

Fig. 6(a) and (b) signify a continued improvement in sulphur reduction compared to the previous treatment duration of 1 hour.

The utilization of benzoyl peroxide as the devulcanizing agent and o-xylene as the solvent for devulcanization process at a temperature of 80℃ for 2 hours, resulted in a 10.5% devulcanization [25]. However, in the process mentioned in this paper, employing sulphur-free toluene as the solvent, achieved a significantly higher devulcanization of 38.09% in just 1.5 hours, highlighting the efficiency and effectiveness of the approach.

The Table 4 provides a concise overview of the sulphur content change due to treatments with o-xylene and toluene over different durations.

Table 4. Elemental composition by weight of Toluene & BPO treated 1.5 hours sample

Solvent	Treatment Time	Initial Sulphur Content(wt.%)	Final Sulphur Content(wt.%)	Sulphur Reduction
o-Xylene	1 hour	2.73%	1.78%	34.79%
o-Xylene	1.5 hours	2.73%	1.56%	42.85%
Toluene	1 hour	2.73%	1.82%	33.33%
Toluene	1.5 hours	2.73%	1.69%	38.09%

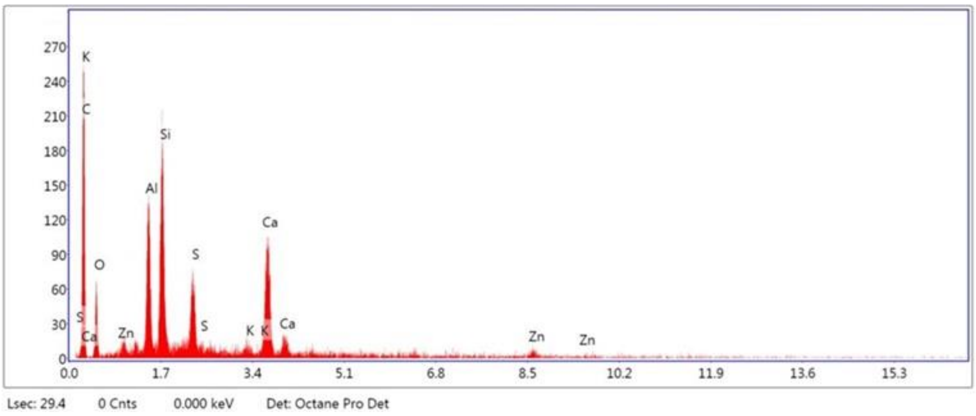

Fig. 4. EDAX Analysis of untreated reclaimed rubber

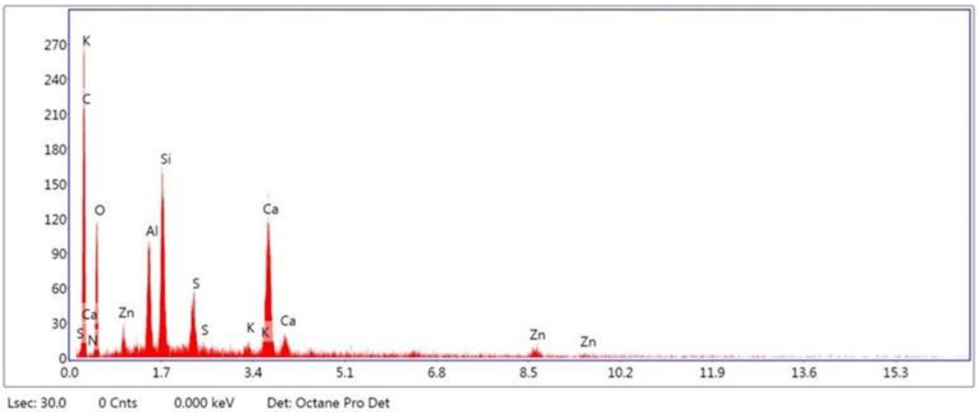

Fig. 5(a). EDAX Analysis of sample treated with o-Xylene & BPO for 1 hour

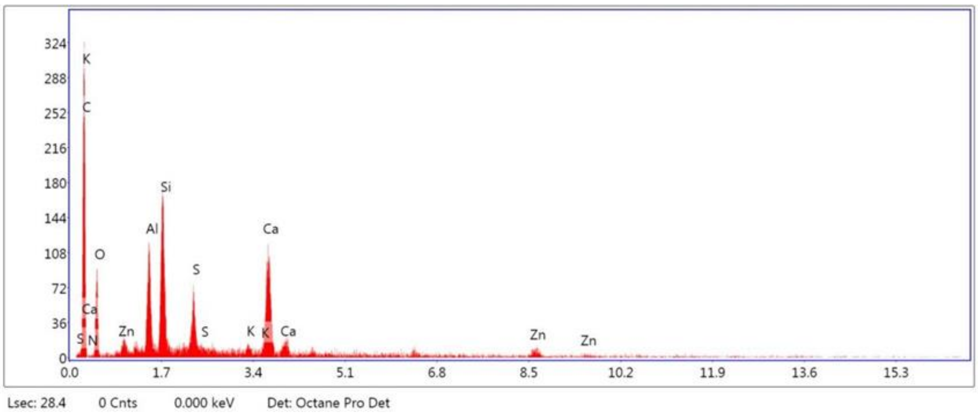

Fig. 5(b). EDAX Analysis of sample treated with Toluene & BPO for 1 hour

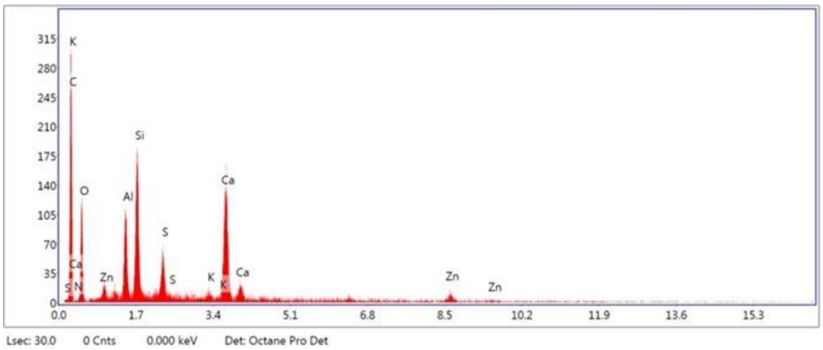

Fig. 6(a). EDAX Analysis of sample treated with o-Xylene & BPO for 1.5 hours

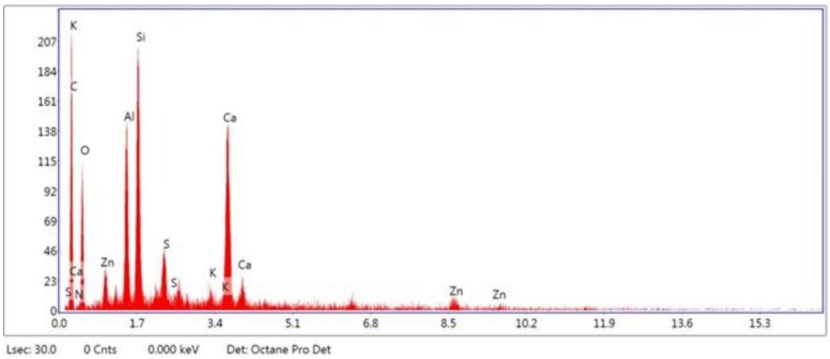

Fig. 6(b). EDAX Analysis of sample treated with Toluene & BPO for 1.5 hours

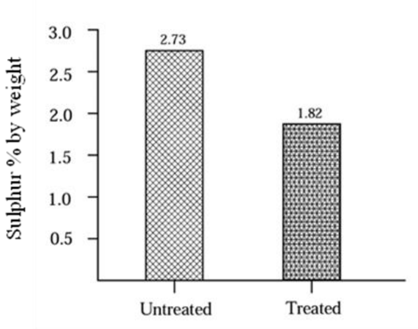

Fig. 7(a). Sulphur reduction of reclaimed rubber treated for 1 hour with Benzoyl Peroxide in Toluene

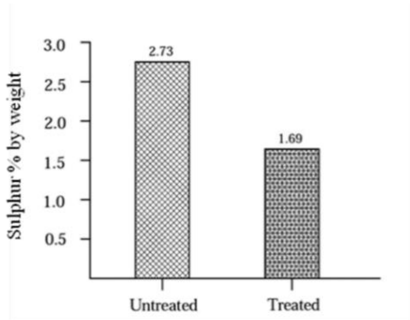

Fig. 7(b). Sulphur reduction of reclaimed rubber treated for 1.5 hours with Benzoyl Peroxide in Toluene

4. CONCLUSION

The devulcanization of reclaimed rubber carried out by using both o-xylene and toluene as solvents, displayed successful breakdown of sulphur cross-links in the rubber samples. The EDAX characterization revealed the elemental composition of the devulcanized rubber, confirming its suitability for potential reuse in various applications. Furthermore, the samples treated for 1.5 hours showed better reduction in sulphur compared to samples treated for 1 hour in both solvents treated with benzoyl peroxide.

The Fig. 7(a)&7(b) illustrate the reduction of sulphur in samples immersed in sulphur-free toluene solvent.

The use of toluene as solvent for devulcanization showed similar results in sulphur reduction as compared to o-xylene. This provides valuable insights into the devulcanization of rubber using chemical solvents, highlighting their potential for waste rubber recycling after blending with virgin rubber and re-vulcanizing, and promoting a more sustainable approach to rubber waste management.

DATA AVAILABILITY

The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of manuscripts.

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CONFLICTS OF INTEREST

The authors confirm that this work is original and has not been published elsewhere nor is it currently under consideration for publication elsewhere. The authors declare that they have no conflict of interest.

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