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Devulcanizing Algerian End-of-life Tire Rubber for Rubber Sustainability and Rubber Product Circular Economy, in Algeria

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ABSTRACT

Managing end-of-life tires (ELTs) remains a persistent global challenge for the transportation sector. Once discarded due to wear or irreparable damage, scrap tires pose severe environmental and health hazards. Although various disposal methods have been developed including landfilling, incineration, and crumb rubber production most are unsustainable and environmentally harmful. The vulcanization process, which transforms raw rubber into durable tire material, significantly hinders recycling efforts. However, recent technological advances offer promising solutions. In Algeria, over 6.2 million registered vehicles in 2020 generate more than six million scrap tires annually, with numbers expected to grow rapidly due to increasing vehicle ownership, shorter tire lifespans, and expanding electric and heavy-vehicle fleets. Without proper management, this waste will accumulate dramatically, exacerbating environmental degradation. Windsor Industrial Development Laboratory has developed an innovative devulcanization technology under the EcoCa™ brand, capable of reversing the vulcanization process effectively transforming used tire rubber back into a reusable form. This breakthrough enables the manufacturing of high-quality engineered rubber products. As a first application, the laboratory has successfully produced and tested passenger vehicle parking blocks made entirely from devulcanized rubber. The proposed four-step approach includes: i. Rubber recovery from scrap tires, ii. Devulcanization of the recovered rybber, iii. Compounding the devulcanized rubber, and iv. Manufacturing green products from recycled rubber. This technology offers multiple benefits: addressing environmental pollution, promoting rubber sustainability and circular economy, conserving natural resources, reducing energy consumption and greenhouse gas emissions, creating jobs, and building local technical expertise in Algeria. Windsor's laboratory seeks industrial and academic partners in Algeria to establish a sustainable local recycling chain and support zero-waste manufacturing practices in the Rubber Industry.

Keywords: Scrap tires; End-of-life tires, Rubber recycling, Rubber devulcanization; Engineered rubber products; Rubber sustainability; Rubber product circular economy.

Graphical abstract

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1. Introduction

Rubbers are elastomers, thermosets (vulcanized), hyperelastic, able to deform substantially to still recover their shapes upon removals of applied loads or deformations. Industrial rubbers divide into i. Natural Rubber, NR and ii. Synthetic rubbers (styrene butadiene rubber or SBR, mostly). The former derives from latex, poly(cis-1,4-isoprene), an unsaturated hydrocarbon product of thousands of vegetation types like the Compositae, Moraceae and Apocynaceae families, but most commercial latex comes from the Hevea brasiliensis tree. Synthetic rubbers are synthesized monomers in reactors, from petroleum hydrocarbons, ethylene propylene diene monomer (EPDM), SBR, chloroprene (CR), and isobutylene (IS) being the most common.

The demand for rubber products grew steadily over the last decade even with a world pandemic, slowness in the economy, and major armed conflicts. Sales of NR reached USD 18.27 billions in 2023, and project to reach USD 30.91 billions by 2031, growing at a CAGR (Compound Annual Growth Rate) of 5.4% between 2024 and 2031. The vast majority of NR (91%) came from Asia Pacific, while Europe, the Middle East, and Africa chipped in approximately 6.5% only. Natural rubber largely contributes to manufacture tires and single-use gloves, which production increased tremendously to meet rising healthcare demands since the recent Covid-19 pandemic. Synthetic rubbers funnel into the Transportation Industry to produce tires.

Rubber scrap is a sizeable component of today's total solid waste. The majority of such scrap derives from tires from automobiles, trucks, off-road vehicles, construction machinery, and motorcycles, but also from other sources including clothing, footwear, gaskets (from oil and gas, household, construction uses), and furniture. In 2024, the United States (U.S.) generated 10.7 million mt of rubber waste, out of which 55% were incinerated for energy recovery, 27% were landfilled, and only 17% were recycled. Recycling rubber waste is challenging, and efforts to improve related policies and technologies continue to evolve to increase rubber sustainability and its circularity.

As most rubber end-up in tires, research on devulcanization targeted waste tires, mostly made of NR and SBR, and common sulfur vulcanization. Figure 1 shows percentages of constituents of car and truck tires in the European Union (EU), with additional data on Table I (including Europe). The paper provides a concise technical overview on the most important devulcanization technologies on sulfur-vulcanized rubbers. Considering the importance to transition to a circular economy, rubber devulcanizate applications concentrated on how devulcanized rubber compound blend with polymeric matrices to develop eco-sustainable polymer materials, mixes, and products.

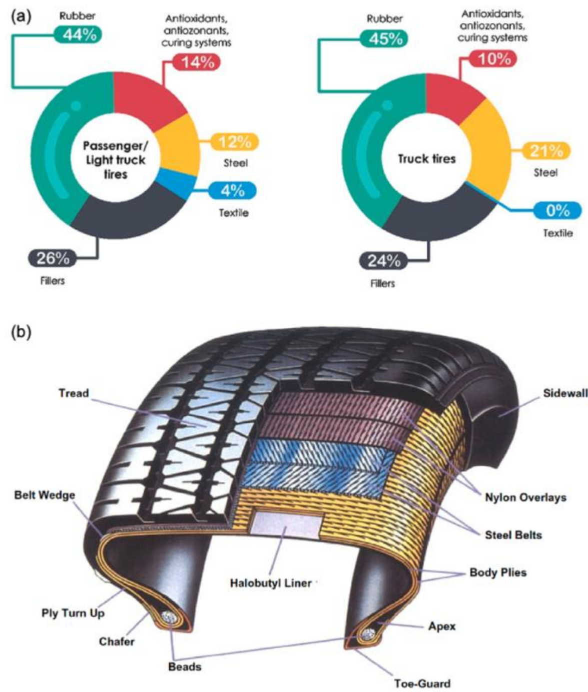

Fig 1. Typical passenger vehicle tire constituents (a) composition (b) design breakdown

Table I. Typical passenger vehicle and truck tire constituents in Europe and the U.S.

Materials	In the U.S.		in the European Union	
	Passenger tire	Truck tire	Passenger tire	Truck tire
Natural rubber (%)	14	27	22	30
Synthetic rubbers (%)	27	14	23	15
Carbon black (%)	28	28	28	20
Steel (%)	14 to 15	13 to 15	13	25
Fabrics, fillers, accelerators, antiozonants, etc. (%)	16 to 17	16 to 17	14	10
Average weight (kg)				
New	11	54	8.5	65
Scrap	9	45	7	56

2. Current Rubber Waste Disposal Pathways

Traditionally, scrap tires ended-up in landfills or burnt to manage landfills (in the open atmosphere, particularly in poorer regions of the world) or in facilities aiming at generating heat or electricity. Figure 2 categorizes additional strategies to cater to retiring tires.

2.1 Primary Tire Valorization

The first revalorization of a scrap tire is its reuse as a used tire after inspection and necessary repairs. Worn tires (below acceptable levels) may undergo retreading. Tire retreading (known as recap or remold as well) is a re-manufacturing method replacing treads on worn but still sound tires. Retreading applies to casings following inspection and needed repairs. This approach preserves almost 90% of the material in each scrap tire fit for retreading for an added 10% in material cost compared to manufacturing a similar new tire.

2.1.1 Worn Tire Retreading

Retreading used tires follows two processes: i. Mold Cure and ii. Pre Cure. Both processes start with tire inspection followed by non-destructive testing (NDT) such as shearography to look for non-visible damage and embedded debris and nails. Select casings deserve mechanical repairing and those damaged beyond repair undergo higher level recycling. Tires with centrally sound casings may retread multiple times. Tires used on short city delivery vehicles retread more than transport tires over their respective service lives. Candidate casings for retreading undergo buffing away and grinding of old, uneven, and damaged treads, in preparation for retreading. This level of revalorization allows tires to remain in service, and saves on the abuse of large amounts of rubbers to make new tires, especially for the large calibers. The cost to retread a tire is some fifth of making a new tire. Large commercial vehicle tires last up to several hundred miles as they retread two to three times as necessary.

Pre-cure. – Pre-extruded tread strips of select profiles cement to tire casings for pre-cure retreading. The method does not need a mold and accommodates varying tire sizes. Therefore, it is widespread, even if a seam results at the junction of each strip upon installation.

Mold cure. – Old, worn, and uneven tire treads of each tire qualified for retreading, undergo buffing away from its casing. Uncured rubber applied around the cleaned tire casing placed in a mold cures into a new tread of a chosen design. A dedicated mold is required for each tire size and tread profile. Mold cure retreading is very similar to that of new tire manufacturing.

Tires damaged beyond repair fill damping sites or undergo recycling or burning.

2.1.2 Scrap Tire Landfilling

Disposals at certified sites accepting waste tires were among the earliest rubber and tire discharge avenues due to the availability of large land surfaces and economic feasibility, but with severe drawbacks. Indeed, landfilled tires can release metals and toxic stabilizers, plasticizers, flame retardants, and other low-molecular-weight additives, which pollute the soil and decrease its fertility. Besides, tire waste collect rain water to become breeding grounds for mosquitoes, rodents, and snakes, sources for deadly diseases, especially in warmer climates and poorer geographies. Piles of tires in open dumpsites or landfills risk flammability, and tire fires often burn for weeks until self-extinguishing, even in advanced societies. Most importantly, landfilling fails to recover useful materials to reintroduce as second-generation feedstock. Therefore, they should be used only when all other waste management technologies (reducing, reusing, recycling, and recovery) cannot be adopted. For all such reasons, the European Commission forbad landfilling end-of-life tires (ELTs) as early as 1999. This has boosted research for alternative options to manage rubber wastes, since.

2.1.3 Secondary Tire Valorization

The second revalorization of a scrap tire damaged beyond repair starts with grinding. The latter involves technologies that reduce the size of waste rubber to powder of several sizes. Common technologies mechanically reducing the size of waste rubber are ambient, wet, and cryogenic grinding.

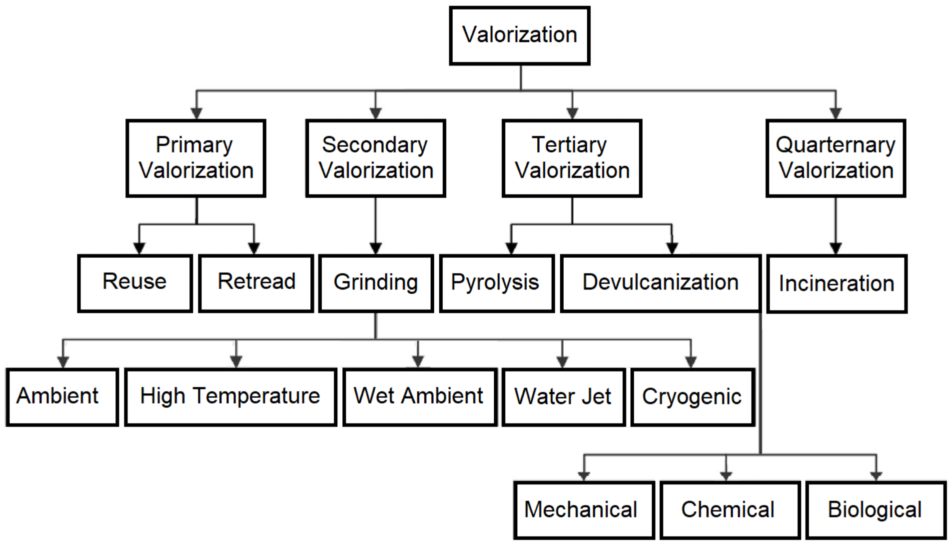

Fig 2. Stages of valorizations of waste tire rubber (WTR)

Tire size reduction produces nugget, crumb, and powder rubbers mostly for civil engineering and manufacturing. Applications include covers in playgrounds, lower layers of floors, walkway tiles, mulch for agriculture and landscaping, sport covers such as running tracks, crash barriers, and shock absorbers, but also as asphalt modifiers and lightweight fillers. Shredded, crumb, and ground rubbers made replacements to gravel, sand, and filler materials, respectively.

Blends of ground tire rubber (GTR) with thermoplastics polyethylene (PE), polystyrene (PS), polypropylene (PP), and polyvinyl chloride (PVC), extrude and mold products, whose physical and mechanical properties depend on properties of the matrix and GTR fraction, particle size, dispersion degree, and GTR-matrix interfacial interaction. Moreover, GTR compounded with virgin rubber produced conveyor belts, shoe soles and heels, molded and extruded profiles, car mats, tubes, sealing plates, mattresses, battery boxes, and other hard rubber goods. However, the direct incorporation of GTR in virgin rubbers usually deteriorates their mechanical and viscoelastic properties, especially the tensile strength and strain at break with the lack of reactive sites on the GTR surfaces or good filler-matrix adhesion. Moreover, GTR can affect the behavior of rubber during curing, as sulfur or accelerators migrate from vulcanized GTR to virgin matrices or vice versa. Still, many researchers included up to 30% of GTR in virgin rubbers without impairing the main mechanical properties of the final products while bringing about advantages like general increases in damping. Besides, the introduction of polar groups addressed poor GTR-matrices adhesion, increasing GTR surface reactivity, using technics classified as physical methods like plasma, ozone, high-energy gamma or ultraviolet irradiation, or as chemical methods involving acids, coupling agents, and chlorination treatments.

Another field of research and development considerably developing over the last decade is embedding GTR in cement, concrete, and asphalt: GTR improves the fracture resistance of concrete, decreases its density, and favors heat, sound, and vibration absorption properties. Blending GTR with asphalt improves the performance and longevity of roads as it reduces vehicle noise, improves crack resistance, and enhances driving comfort. Finally, in sludge treatment plants, a bed of GTR can absorb organic solvents, mercury (II), and other heavy metals, due to the presence of thiol and other sulfur residues able to immobilize metal ions.

2.1.4 Tertiary Tire Valorization

A process seeing widespread use, for simplicity, and to avoid accumulating scrap tires, is incineration. The process is highly exothermic, initiating at controlled high temperatures (in the 1,000 C) to become self-sustaining. If temperature remains high enough, the process only yields water (H_2O), oxygen (O_2), and carbon dioxide (CO_2), while too low processing temperatures lead to the emission of several toxic gases, such as dioxins or persistent organic pollutants (POPs).

In fact, the Tire Industry recourses to incineration to dispose of production waste and rejects and produce plants' own energies. It is also a common process in cement kilns, steel mills, thermal power plants, pulp and paper mills, industrial boilers, and sewage treatment installations. For cement manufacturing, high temperatures (in the range of 2,000 C) ensure complete combustion of tire components, converting steel to iron oxide and sulfur to sulfates, useful ingredients in the final cement products.

2.1.5 Scrap Tires Pyrolysis

Pyrolysis decomposes the organic fraction of waste tires at high temperatures (400 to 1,200 C), under reducing or inert atmospheres, generating solid, liquid, and gaseous products. The solid carbon-rich residue purified from steel, fiberglass, and contaminants, produces activated carbon, recovered carbon black (CB), and regenerated inorganic fillers. The liquid tar, water, aromatic hydrocarbons, and organic substances, makes for a fuel after sulfur removal. The gaseous products, rich in methane, ethane (C_2H_6), ethylene (C_2H_4), propylene (C_3H_6), butylene (C_4H_8), carbon dioxide (CO), and carbon monoxide, also produce energy. Although pyrolysis saves fuel, it is less industrially widespread than incineration, for investments in infrastructures, low purity of obtained products, and high operating and maintenance costs.

An equivalent process to pyrolysis is gasification or the partial oxidation employing pressure, heat, and a reactive agent such as air, oxygen, hydrogen, or steam to convert tire waste into syngas, primarily composed of CO, H_2, CO_2, and light hydrocarbons (methane, CH_4), energizing cells and turbines. Advantages of gasification include high conversion and energy efficiency (about 34% more). Besides, syngas contaminants (sulfide, H_2S and ammonia, NH_3) are manageable. Still, gasification involves higher temperatures (1,200 to 1,500 C) than pyrolysis.

2.1.6 Scrap Tires Rubber Devulcanization

Devulcanization of post-consumer rubber the bulk of which being ELTs is most desirable to rubber waste management. It converts a three-dimensional, interlinked, insoluble, infusible thermoset polymer network to an essentially thermoplastic like material, while ideally keeping the same properties of virgin rubber to then process and revulcanize into new products. Hence, rubber devulcanization is the most promising technology to tackle the issue of waste rubber and at the same time save natural resources for future generations.

The first attempts at devulcanizing scrap rubber goes back to more than a century ago. Since, many devulcanization technologies developed, reported on Figure 3, broadly classify into three main groups: i. Physical, ii. Chemical, and iii. Biological. Physical technics consist of mechanical, thermo-mechanical, microwave-based, and ultrasonic, chemical processes involve chemical agents, whereas biological devulcanization involves microorganisms to break rubber crosslinks.

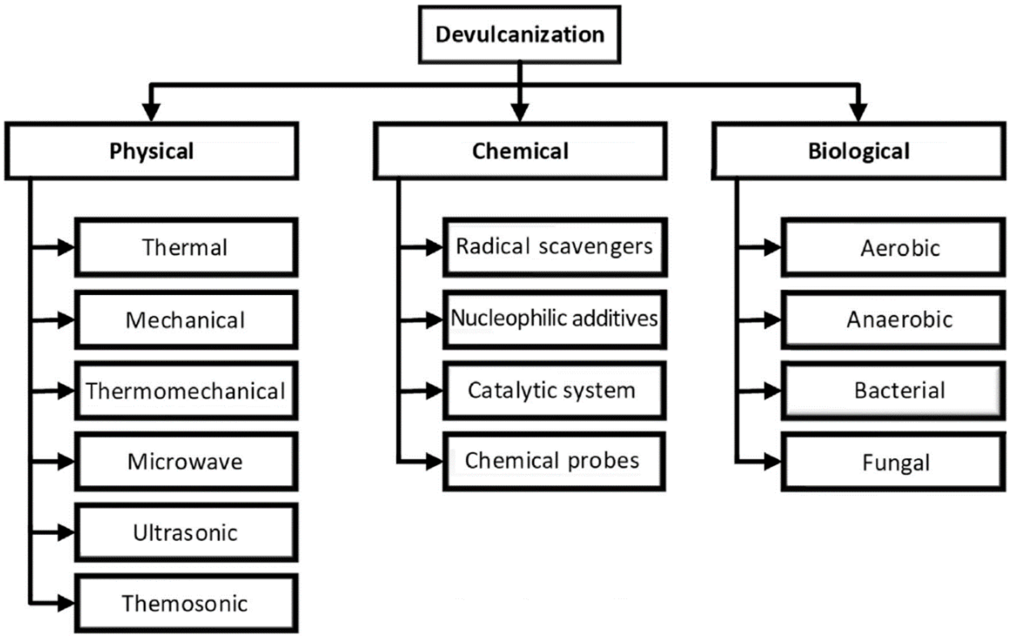

Fig 3. Devulcanization technologies for vulcanized rubber

Any procedure in which vulcanized rubber waste transforms for mixing with virgin rubber, processing, and revulcanizing, is called reclaiming or reclamation. This has a high potential to recycle rubber waste products, as it allows breaking three-dimensional networks to decrease molecular weight to achieve plasticity and reprocessing of scrap rubber. However, reclaiming cleaves carbon-carbon (C-C) bonds on rubber backbone degrading initial macromolecules. Therefore, devulcanization is more desirable, to selectively cleave intermolecular sulfidic carbon-sulfur (C-S) and/or sulfur-sulfur (S-S) bonds, breaking down the spatial network of vulcanized rubber while avoiding main chain scissions and degradation of the polymer [1].

Rubber Mechanical Devulcanization

Thermo-mechanical devulcanization is the most widely used in the industry. Rubber crumb undergoes mechanical shearing at about 200 C, brought by internal friction and from outside sources. Solvents like water, oils, hexane, or supercritical fluids added before or during grinding, facilitate the process, solubilize the small chains, and swell the rubber.

Thermo-mechanical devulcanization, largely studied during the last decades, generally yields high devulcanization throughputs. The most common mechanical and thermomechanical devulcanization technologies use batch mixers or continuous extruders, besides other equipment.

Batch Mixers. Devulcanization based on batch mixers is relatively simple, low-cost, and environmentally friendly, and is generally performed without external heating or chemicals. Batch mixers divide into: i. Open, two-roll mills or Brabender mixers, and ii. Closed, Lancaster-Banbury or internal batch mixers. Crumb rubber devulcanizes by intense mechanical shearing for minutes, with temperature reaching 250 C. To prevent excessive heating and degradation of polymer chains, a recent technique proposes a water-cooled two-roll mixing mill, to devulcanize CB-filled NR. However, the resultant degree of devulcanization remained low, between 20 and 37.8%.

Extruders. The most common mechanical devulcanization is Ficker's or single and twin-screw extruding. The technology is widely used as extruders are commonly and readily available in rubber manufacturing and allow for continuous high-yield devulcanization. Optimizing screw speed and barrel temperature for proper devulcanization correlate processing parameters with devulcanization quality and yield, and surface morphology. Crosslink scission selectivity decreases as temperature increases, with a consequent increase in C-C bond cleavage and reduction in mechanical performance of the revulcanizate. Therefore, the quality of devulcanization increases at moderate operating conditions.

Other Approaches. Researchers devulcanized waste rubber in a unique metallic-cone-like high shear mixer, water-cooled, with one cone static while the other rotates and pressurizes the rubber to devulcanize. Large chunks of waste rubber feed the instrument for size reduction and decrease in crosslink density. However, such reactions mainly occur at the rubber particle surface, classifying this technique as a rubber surface activation rather than a bulk devulcanization.

Another devulcanization technology is the High-Pressure High Temperature Sintering (HPHTS), which allows recycling vulcanized rubber powder by applying heat and pressure. Pressure compresses the particles and increases the inter-particle contact, while temperature (between 80 and 240 C) promotes cleavage of crosslink bonds. This in turn allows the formation of new covalent bonds at particle interfaces, thereby sintering the particles into a single piece. Researchers found that the mechanical properties of sintered rubber compare with those of conventionally manufactured rubbers.

Thermo-mechanical devulcanization may involve different devulcanizing agents, to promote further scission of sulfidic bonds, for mechano-chemical devulcanization.

Ultrasonic Devulcanization. Ultrasonic devulcanization is one of the most convenient, as it allows a high degree of devulcanization and precise control of the properties of devulcanizates. This type of devulcanization is fast and does not involve chemical solvents. However, it requires specialized technological equipment, which hinders to this day its large-scale application.

Ultrasonic devulcanization methods exploit mechanical waves at high frequencies to produce tensile-compressive stresses and cavitation bubbles in the medium, providing enough energy to break C-S and S-S bonds. However, under pressure and temperature, ultrasonic waves could also break carbon backbone chains, so researchers stress on selecting the proper test conditions.

Microwave Devulcanization. Microwave devulcanization uses irradiations to energize molecules, thereby raising temperature and inducing crosslink breakage. However, while microwave processes are hard to implement along a production process, they remain among the most widely investigated devulcanization technics.

Supercritical Devulcanization. A supercritical state is a particular aggregation coexisting gas and liquid phases. Because of the absence of liquid/gas phase boundaries, supercritical fluids (SCF) have no surface tension, show solvent properties similar to those of liquids, and transport properties similar to those of gases. Therefore, they can dissolve solutes, are miscible with ordinary gases, and can penetrate pores in solids. Supercritical fluids show liquid-like density, but their viscosity and diffusivity are intermediate between those of gases and liquids.

Supercritical carbon dioxide ($scCO_2$) appeared recently as an innovative and “green” devulcanization medium, due to its chemical inactivity, non-toxicity, non-flammability, and low cost. Its low-temperature low-pressure critical points (of about 31.1 C and 7.4 MPa) allow the use of simple equipment, and excess $scCO_2$ can easily release to the ambient. The role of $scCO_2$ is yet to understand, but it probably assists in swelling vulcanized rubber, stretching the sulfide links and allowing devulcanizing agents to penetrate into rubber to devulcanize.

Rubber Chemical Devulcanization

Chemical devulcanization, applied since the 1960s, is among the most widespread. It employs organic and inorganic chemicals that selectively break C-S and/or S-S bonds. Generally, a supply of thermal and mechanical energy accelerates the treatment. Chemical devulcanization uses batch processes, mostly, in which ground waste rubber mixes with chemical agents, at a controlled temperature and pressure. Chemicals used include sulfides, mercaptans, amines, inorganic solvents like propane thiol/piperidine, triphenylphosphine (PPh_3), trialkyl phosphites, lithium aluminum hydride, and methyl iodide, organic solvents like alcohols and ketones, and ionic liquids (ILs). The drawback of chemical devulcanization relates to the toxicity of chemicals. The process could employ less toxic, more environmentally friendly, non-sulfured agents, some of which are yet to diffuse.

Sulfides and Mercaptans. The most widely used chemicals in rubber devulcanization are sulfides and mercaptans such as disulfides (DD, thiophenols, and their zinc salts), thiol-amine reagents, hydroxide or chlorinated hydrocarbons, added typically in concentrations of 0.5 to 10 wt%.

Amine-based Devulcanization. Van Duin et al. patented amine-based devulcanization in 2003. Their work showed amines to help high temperature radical devulcanization. They preferred 0.1 to 15 wt% of amine compounds to work with α-H atoms to reduce the crosslink density by scission. Sulfides and mercaptans (amine-based chemicals) often combine with other devulcanization treatments. Dijkhuis et al. employed hexadecylamine (HDA) as an agent for EPDM thermal devulcanization and reported reduction in crosslink density of 50% at 225 to 275 C, even though di- and poly-sulfide bonds cleaved. Devulcanizates blended with virgin EPDM showed good mechanical properties compared to virgin vulcanizate. Hexadecyl- and other amines like tetraethylenepentamine (TEPA) also combined with ultrasonic mechanical devulcanization, allowing treatment at lower temperatures.

Other Compounds. Other catalysts, inorganic and sulfur-free organic developed in the past decades. Some are propane thiol/piperidine, Grubbs catalysts, PPh3, trialkyl phosphites, lithium aluminum hydride, methyl iodide, 1,8-diazobicyclo [5.4.0]undec-7-ene (DBU), and benzoyl peroxide (BPO). Another organic solvent, 2-butanol, suits between 200 and 350 C. To decrease process temperature and increase eco-sustainability of devulcanization, 2-butanol partly substitutes turpentine liquids particularly α-terpineol, derivable from renewable resources. Deep eutectic solvents (DES) like mixtures of choline chloride (ChCl) or $ZnCl_2$ with urea, p-toluene sulfonic acid, or glycerol, rose as devulcanization agents, especially in combination with ultrasonic processes. These solvents are widely tunable, non-flammable, and have low volatility and toxicity.

Ionic Liquids. Phosphonium, imidazolium, and pyrrolidinium ionic liquids (IL) salts also suit physical and chemical devulcanization due to high conductivity, high thermal stability, low flammability, and low volatility. Not only are ILs safer and less toxic than other solvents, they can solubilize a large variety of compounds and their properties are tunable to selected cations and anions. Ionic liquids, alone or in combination, proved effective on NR like trihexyl(tetradecyl)phosphonium chloride or N,N-dioctylimidaolium bromide combined with Grubbs catalyst, in producing telechelic oligomers such as acetoxy telechelic polyisoprene with high (95 to 99%) yield.

Rubber Biological Devulcanization

While vulcanized materials are resistant to microbial attack, it is possible to promote biological devulcanization, with bacteria and fungi selectively breaking sulfur bonds in rubbers. However, biotechnological devulcanization processes did not apply industrially, for persistent limitations in devulcanization ratios, bacteriological contamination risks, and limitations to surfaces of components.

The scientific literature contains various examples of bacterial devulcanization on rubber substrates such as GTR, NR, and latex [1]. The process, in aerobic or anaerobic, proved feasible with several strands of bacteria such as Sphingomonas, Alicyclobacillus, Gordonia desulfuricans, Nocardia, Rhodococcus, and Bacillus cerus. For anaerobic bacterial devulcanization, researchers used sulfur-reduction bacteria, while in aerobic devulcanization sulfur oxidizes produce sulfone groups on rubber surfaces. Reports indicate 1 to 30 days, at 30 C, to decrease sulfur content in a sample of 8 to 30%, resulting in a partly devulcanized product to revulcanize for improved mechanical properties compared to those of virgin rubber.

Another type of biological devulcanization uses fungi. Fungal devulcanization on three white-rot fungi (i.e., Pleurotus sajor-caju, Trametes versicolor, and Recinicum bicolor) degraded Poly-R478. The study showed Recinicum bicolor of highest efficiency and growth rate proportional to sulfur oxidation rates in the rubber matrix. In another study, a white rot basidiomycete, Ceriporiopsis subvermispora, degraded vulcanized NR sheets on a wood medium. The fungus promoted cleavage of sulfide bonds and decreased sulfur content in rubber by 29% in 200 days, thus a potential of ligninolytic basidiomycetes for rubber biological devulcanization.

Despite increased research on rubber biological devulcanization, the commercial exploitation of this technology is currently only performed by few companies on a small to medium scale. In particular, the Recircle group in England (https://www.recircle.com/) applies bacterial devulcanization on natural and synthetic ground rubbers, yielding intermediates, to revulcanize.

2.2 Quaternary Tire Valorization

A basic rubber waste management consists of using scrap rubber as fuel (known as TDF or tire-derived fuel). Tires are over 90% organic and have a calorific value of 32.6 MJ/kg, competing with coals (gross calorific value of about 23.9 MJ/kg on an ash-free but moist basis). Approximately 45 and 38% of post-consumer tires and industrial wastes in the U.S. and in the EU, are used as supplementary non-fossil fuel forms of energy recovery, including incineration, pyrolysis, and gasification.

3. Recent Developments in Rubber Devulcanization

Çataklı and Erguder [2] focused on a new approach to valorize GTR and nitrate-containing wastewater via simultaneous devulcanization and denitrification. They investigated biological devulcanization (biodevulcanization) of GTR with enriched cultures. Görbe et al. [3] developed a thermomechanical process of GTR by incorporating chemicals to achieve better devulcanization. Samples devulcanized in a twin-screw extruder with three chemicals: N-(cyclohexylthio) phthalimide, dibenzamid diphenyl disulfide, and dialkyl-pentasulfide (DPS, commercialized as Aktiplast 79). Rodak et al. [4] used CB N220 and N660 as reinforcing fillers for SBR blended with reclaimed GTR. They investigated combined effects of GTR devulcanization level and CB grade on properties of SBR/GTR composites considering curing characteristics, thermal stability, mechanical properties, dynamic behavior, swelling, and morphology. Parsamanesh et al. [5] reviewed progress in rubber and plastic recycling technologies. They reported thermomechanical devulcanization of butyl rubber, bromobutyl rubber, and a waste commercial butyl rubber using a twin-screw extruder.

Roetman et al. [6] focused on identifying opportunities and barriers for innovation through devulcanization. They interviewed 36 European organizations, identified to develop and utilize devulcanization to transform rubber from end-of-life tires into resources for new rubber products. Lewandowski et al. [7] investigated the role of cryogenic grinding and devulcanization on tensile and elastocaloric properties of NR and GTR. Microwave irradiation on crosslinks breakage and rubber chain scission used Fourier transform infrared (FTIR) to show decreases of S-S, C-S, and C-C bonds. The NR/GTR blends high elastocaloric effect compared to pristine NR with CB. Colom et al. [8] combined FTIR, spectroscopy, and derivative thermogravimetric analysis to assess structural, physical, and thermal behaviors of GTR samples devulcanized by microwaves and a combination of a thermal-chemical-mechanical process involving a devulcanization aid benzoyl peroxide and microwaves.

Danila and Januševičius [9] compared adsorption of Pb(II) ions from an aqueous solution using non-devulcanized and devulcanized tire rubber powder from used truck tires through mechano-chemical devulcanization. Characterization used Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy, SEM–EDS, Fourier Transform Infrared Spectroscopy, FTIR, and X-ray Diffraction, XRD analyses. Different adsorption isotherm and kinetic models analysed adsorption mechanisms. Colom et al. [10] examined samples made from NR, SBR, and nitrile butadiene rubber (NBR), blended with proportions of devulcanized GTR (dGTR) and newly revulcanized rubber, along with silicon dioxide or silica (SiO_2), commonly present in tire formulations. Sample treatment and the influence of SiO2, with the presence of a silane-based agent (TESPT) promoting the interaction between rubber and silica, were analyzed at the microstructural level (using FTIR, Thermogravimetric Analysis, TGA, SEM) and through mechanical testing. Ghosh et al. [11] transformed

GTR into elastomeric composites through optimized devulcanization and blending with fillers like CB and silica-silane. Effects of silane concentration on tensile strength, Payne effect, and dispersion enhanced polymer-filler interaction through improved silanization.

Kędzia et al. [12] explored the potential for recycling NR latex waste from balloon production through devulcanization and revulcanization. Mechanical devulcanization of colored latex balloon waste preceded revulcanization using a sulfur-based system. The reclaimed rubber crosslink density, tensile strength, and abrasion resistance, compared with those of virgin NR. Hardness and abrasion resistance compared too, indicating successful material recovery. Structural analyses, including FTIR and SEM, revealed devulcanization to allow for successful revulcanization. Obukhova et al. [14] developed a method of directed thermomechanical devulcanization, to ensure solubility of CR in polydisperse composite, preventing the formation of aggregates consisting of unsaturated CR particles, whose elastic aftereffect causes intensive cracking, especially during low-temperature road operations. Kohári and Bárány [15] prepared blends of thermoplastic polyurethane (TPU), GTR, and dGTR. They aimed at producing thermoplastic elastomers (TPE) with TPU partially substituted by GTR or dGTR obtained from used tires, thereby forming a blend with a favorable cost/value and smaller environmental footprint. Akkenzheyeva et al. [16] investigated the use of rubber-polymer composites ELTC (End of Life Tire Compound) in bitumen. They suggested ELTC for plastics such as PE and PP. They obtained ELTC by rubber devulcanization at relatively low temperatures.

Colom et al. [17] investigated devulcanization of GTR from car and truck tires subjected to varying periods of microwave irradiation. The devulcanized GTR was then blended with NR to undergo vulcanization, simulating recycling for novel applications. Hejna et al. [18] thermos-mechanically treated GTR in a twin-screw extruder with zinc borate, to enhance shear forces. They then introduced modified GTR into flexible polyurethane (PU) foams, and investigated the impact of modification parameters on the cellular structure, static and dynamic mechanical performance, thermal stability, thermal insulation, and acoustic properties. Rosales et al. [19] explored the viability of obtaining a completed recycled composite with improved toughness by adding GTR to linear low-density polyethylene (LLDPE)/PP blends. The idea of maintaining additional processing steps low and reducing costs prevailed. Material evaluation relied on mechanical and fracture behavior, in relation to microstructure and composition. Ghosh et al. [20] conducted thermo-mechanical-chemical devulcanization using six potential devulcanization aids (DAs). They chose silanes, widely used in tire rubber, as coupling agents for silica. They reported network breakdown, miscibility of the devulcanizate, and mechanical properties of devulcanized and revulcanized rubbers.

Mondal et al. [21] looked at devulcanizing agent availability, low cost, smooth penetration in crosslinked networks in light of carrier, multifunctional activity, and eco- friendliness. They focused on GTR devulcanization by way of a multifunctional bis(3- triethoxysilylpropyl)tetrasulfide. The agent devulcanizes GTR mechano-chemically and facilitates homogeneous silica dispersion into the revulcanized rubber. Yazdani et al. [22] investigated the relationship between microstructure and physio-mechanical properties of devulcanized rubber samples. They performed mechanical and identification tests including FTIR and Nuclear Magnetic Resonance, NMR, on samples. Troyano-Valls [23] completed a PhD degree at Massachusetts Institute of Technology (MIT) in which her group further optimized a single component adhesive to produce rubber composites containing 40 wt% recycled rubber crumb and an unused polybutadiene matrix. Innes et al. [24] used micromechanical models for filler reinforcement to evaluate whether recycled rubber behaves elastically. Furthermore, mechanical devulcanization of GTR by solid- state shear milling produced a recycled rubber powder elastically effective when blended with virgin NR. Cataldo et al. [25] mixed a CB-filled and natural rubber-based formulation with different levels of sulfur and studied differential scanning calorimetry, DSC, determination of vulcanization enthalpy.

Magagula et al. [26] reviewed papers using keywords such as “waste tire rubber”, “waste tire pollution”, and “waste tire applications” from 2012 to 2023. Recycling publications have surged by 80% in the past decade, with China and the U.S. leading the research. Pyrolysis and devulcanization emerged as key circular economy advancements, producing fuel and reusable rubber. They quoted 1.5 billion waste tires to accumulate yearly, worldwide, with projections to increase by 70% in the next 30 years if unaddressed. They reported 26 million mt of used tires generating annually worldwide, of which civil engineering and backfilling used 17 million mt in particles. Tires are complex polymer composites, primarily composed of natural and synthetic rubbers. Raslan et al. [27] discussed the partial replacements of waste tire rubber, WTR, and microwave devulcanized rubber at different ratios on the properties of virgin SBR as one of the most essential components of synthetic rubbers in tire production. Fixed percent of tetramethylthiuram disulfide and spindle oil added to WTR exposed to different microwave times. Ketov et al. [28] discussed WTR cracking with organic solvents in rubber devulcanization and dispersion to nano-sized particles. Various practical solutions proposed the resulting modified bituminous binders in road construction and strengthening soil foundations.

Bakir et al. [29] reported cryogenically-ground micron-sized rubber particles with satisfactory physical performance measures in reclaimed rubber-based tires. The chemically functionalized cryogenically ground micron-sized rubber particles possess reactive silica particle sites ultimately designed to facilitate the participation in post-polymerization with a host matrix allowing higher loading levels than state-of-the-art configurations. Abdullah [30] proposed thermomechanical devulcanization to restore formability of waste rubber for recycling, then mixing with chemical agents and hot rolling and extrusion to form rubber sheet products. Chouchaoui proposed the same for any rubber manufacturing [31]. Susik et al. [32] thermo-mechanically treated GTR in the presence of styrene-butadiene-styrene, SBS, copolymers. Subsequently, GTR modified by SBS copolymer and crosslinking agents (sulfur-based and dicumyl peroxide) underwent characterization for rheological, mechanical, and morphological properties.

Rodak et al. [33] prepared GTR and SBS block copolymer blends at 50/50 wt%, with the application of four SBS copolymer grades (linear and radial) and two crosslinking agents (a sulfur-based and dicumyl peroxide), by melt compounding. Characterization assessed rheology, crosslinking, mechanical parameters (i.e., tensile properties, abrasion resistance, hardness, swelling, and density), thermal stability, and morphology of prepared materials. Leong et al. [34] overviewed the legislative frameworks, techniques, challenges, and trends of rubber waste management in various countries. The 4R (reduce, reuse, recycle, and recover) framework applied in waste management appeared to be viable for the processing of rubber waste. Certain countries especially of the EU implemented extended producer responsibility (EPR) to manage collection of rubber waste, particularly used tires. Leong et al. [34] discussed the processing of rubber waste in each level of the 4R hierarchy, with detailed elaboration on the most practiced 'R', recycling via physical and/or chemical means. They highlighted challenges faced in implementing rubber waste management in different countries and provided recommendations for a more sustainable rubber consumption.

Cheng and Wang [35] introduced traditional internal mixing combined with a custom-built ultrasonic generator. They investigated effects of ultrasonic parameters on the performance of tread rubber formulations. Olszewski et al. [36] investigated the structure and performance of PU/GTR composites differing in isocyanate index (from 0.8 to 1.2) and prepared with and without considering the GTR functionalities in formulation development. Bianchi et al. [37] highlighted the use of high-speed thermokinetic mixing as an alternative to recycling GTR using mechano-chemical treatment. Wiśniewska et al. [38] showed reactive extrusion promises for rubber devulcanization. They presented state-of-the-art in patented WTR devulcanization including dynamic desulfurization, reactive extrusion, microwave treatment, and other less popular methods. Special attention focused on used components, rubber treatment conditions, and static mechanical properties of reclaimed rubbers. Pérez-Campos et al. [39] used a resonant technique based on a dual-mode cylindrical cavity to simultaneously-heat rubber and measure its permittivity. Kirshanov et al. [40] proposed a new method to recycle polyester tire cord under oligoethylene terephthalates, bis(2-hydroxyethyl) terephthalate and ethylene glycol. The method involves simultaneous homogeneous glycolysis of polyethylene terephthalate and devulcanization of CR. Kirshanov et al. characterized polyester cord and glycolysates by FTIR, spectroscopy, and gel permeation chromatography (GPC).

Gumede et al. [41] discussed thermo-chemical devulcanization of waste tires in $scCO_2$ using common organic devulcanizing agents. Wang et al. [42] presented high-quality GTR production using $scCO_2$ jet pulverization. They incorporated diphenyl disulfide as a devulcanization reagent. They investigated effects of rubber swelling on particle size distributions, shape characteristics, thermal degradation, and devulcanization of acquired GTR. Kalin [43] reported a patented scalable rubber devulcanization manufacturing process to recover high quality rubber from waste tires on an industrial scale. Li et al. [44] evaluated the compatibility of rubberized asphalt including morphology observation, segregation, rheology, and interaction measurements. They also summarized and compared state-of-art de-crosslinking technologies of GTR for compatibility improvement of rubberized asphalt. Ma et al. [45] proposed silanized silica from waste GTR by thermo-oxidative reclamation. They used a stepwise separation of reclaimed silanized silica from WTR using thermo-oxidative reclamation, and quantified the interfacial interaction between silanized silica and rubber. Ruikun et al. [46] presented an asphalt modifier by miscibility of waste cooking oil (WCO) and rubber. They suggested GTR and WCO to solve segregation of rubber asphalt and low recycling rate of WCO.

Elnaggar et al. [47] prepared SBR and SBR/devulcanized WTR (dWTR) using mechano-chemical and ultrasonic devulcanization of WTR reinforced by polyester tire cord with different contents. Wiśniewska et al. [48] performed low-temperature extrusion of GTR as a pro-ecological waste tires recycling method. They modified GTR with constant content of dicumyl peroxide and variable amounts of elastomer. Xiao et al. [49] suggested recycled rubber aggregates as fillers and modifiers in producing rubberised asphalt and concrete. They also suggested GTR processing through chemical, mechano-chemical, microwave, ultrasound, and microbial devulcanization to improve its usability in asphalt.

Zedler et al. [50] briefly reviewed GTR functionalization strategies as a promising approach for the production of sustainable adsorbents of environmental pollutants. They showed reactive extrusion as a promising method to develop further GTR-based adsorbents dedicated to environmental pollutants. Krasnovskikh et al. [51] reported poor cosite of rubber with bitumen limiting the use of waste tires rubber for asphalt modification as an environmentally friendly solution. They proposed joint pyrolysis of rubber with oxygen-containing oil under pressure for conversion into a nano-structural bitumen modifier. They studied the proposed product by thermogravimetry, NMR-spectroscopy, chromatomasspectrometry, SEM, and solubility in toluene. Liu et al. [52] prepared TPE based on highly filled LLDPE with GTR (50–90%) via dynamic vulcanization. They quantified interactions between the rubber and plastic by a series of characterizations including mechanical tests, rheological measurements, DSC, dynamic mechanical analysis and scanning electron microscopy.

Karabork et al. [53] evaluated mechanical and anti-corrosion of epoxy coatings modified by recycled waste tire products. They prepared epoxy coatings reinforced with GTR, dGTR, pyrolytic CB, and original CB particle contents for application to the galvanised steel substrate. Micro-mechanical tests (nano-hardness and micro-scratch) allowed measuring the mechanical properties of the coatings. Tribological and corrosive performance of the coatings evaluations used reciprocating ball-on disc and salt spray corrosion tests. El- Nemr et al. [54] replaced virgin SBR by dWTR for preparing composites at fixed content 40 phr (part per hundred part of rubber). Waste silicate treatment used (3- aminopropyl) trimethoxysilane and blending with PS. Mechanical properties measurements for the composites relied on ultimate tensile strength, elongation at break, tensile modulus, and the cross- link density relied on mechanical studies. Application for floor tiles included compression set and abrasion resistance measurements. All results indicated an enhancement in ultimate tensile strength, modulus, and cross- link density by adding silicate fillers. Żukowska et al. [55] incorporated GTR into a flexible foamed PU matrix as a cost-effective waste-based filler. They applied a two-step prepolymer in foam manufacturing to maximize formulation changes. They investigated GTR content impact on foam processing, chemical and cellular structures, and static and dynamic mechanical properties, thermal stability, sound suppression, and thermal insulation.

Fazli and Rodrigue [56] investigated the effect of interfacial interaction and processing (one- and two-step) on the structure-property of ternary blends. The addition of ethylene-vinyl acetate (EVA) and ethylene octene copolymer (POE) aimed at homogenizing structure and improving rubber-like properties of recycled high-density polyethylene (rHDPE) and regenerated tire rubber blends. Wei and Xin [57] investigated surface characteristics of rubber powder modified by deep eutectic solvents using infrared spectroscopy, sol fraction, solid surface energy, and SEM. They studied mechanical properties of modified GTR (mGTR)/SBR/silica composites. They characterized viscoelasticity of the composites by Payne and Mullins effects. Kosmela et al. [58] thermo-mechanically modified GTR particles by adding fresh and waste rapeseed oil in reactive extrusion. Makoundou et al. [59] overviewed existing surface treatments of polymers especially rubber, including gamma ray, ultra-violet-ozone, microwaves, and plasma. Several studies indicated overall improvement of rubber surface reactive properties due to contaminant removal or roughness enhancement attributed to crosslinking or scission reactions occurring on rubber surfaces. Asphalt mix phase stability increased with pre-treated rubber incorporation.

Zedler et al. [60] investigated conventional sulfur and two organic peroxide (dicumyl peroxide and di-(2-tert-butyl-peroxyisopropyl)-benzene) curing systems to tailor the performance of GTR/NBR blends reinforced with highly dispersive silica. They investigated curing characteristics, mechanical and acoustical properties, swelling, thermal stability, and microstructure of the prepared composites. Wiśniewska et al. [61] mixed GTR with dicumyl peroxide and a variable content of ethylene-vinyl acetate copolymers characterized by different vinyl acetate contents. They used auto-thermal extrusion in which heat generated during shearing inside the barrel. They evaluated the processing, performance properties and storage stability of modified reclaimed GTR based on specific mechanical energy, infrared camera images, an oscillating disc rheometer, tensile tests, equilibrium swelling, gas chromatography combined with a flame ionization detector, and gas chromatography with mass spectrometry. Marín-Genescà et al. [62] recycled GTR by separating the fraction of vulcanized rubber from other compounds to later grind and separate into lower particle sizes. They incorporated GTR particles as a filler in EPDM and tested the composite against neat EPDM. Simon and Bárány [63] thermomechanically devulcanized GTR in a co-rotating twin-screw extruder at different barrel temperatures and screw speeds. They measured soluble content and crosslink density of samples in Horikx's graphs.

Alonso Pastor et al. [64] studied the evolution of microstructure of rubber from ELTs during granulation, grinding, and devulcanization. They analyzed different morphologies (particle size distribution and specific surface area) obtained by cryogenic and water jet grinding, and different devulcanization techniques (thermo-mechanical, microwave, and thermo-chemical). Wang et al. [65] detailed the interactions between WTR and high-pressure water jet as a sustainable reclamation technique for WTR. Guo et al. [66] presented an environmentally friendly agent, terpinene, to swell rubber crosslink structures at low temperatures. They conducted rubber swelling and reclaiming experiments with a mechano-chemical devulcanization method to explore swelling of terpinene. Allan et al. [67] investigated the suitability of two sol fraction measurements to assess devulcanization performance of ferrooxidans and a sulfur-oxidising consortium on industrial GTR. Hassan et al. [68] implemented a self-designed thermo-oxidative reactor for exfoliation of CB from GTR, under thermo-oxidative reclaiming without additives. The exfoliation of CB from rubber vulcanizate consisted of scission of main chains and crosslinked network. They discussed the degree of scission by gel permeation chromatography and Horikx theory.

Araujo-Morera et al. [69] reported a successful combination of cryogenic grinding with a chemical treatment for modifying the surface of GTR. Various cryogenic grinding protocols and chemical treatments with different acids led to an optimal particle size and surface modification. Araujo-Morera et al. added mGTR to SBR. Markl and Lackner [70] addressed established devulcanization technologies and novel processes described in the scientific and patent literatures. Buitrago-Suescún and Oscar [71] treated waste GTR, combining oxidation with potassium permanganate/hydrogen peroxide, followed by microwave exposure in the presence of a devulcanizing agent. They analyzed dGTR by FTIR spectroscopy, DSC, TGA, crosslink density and sol fraction.

Halász et al. [72] studied the effect of various rubber formulations, processing conditions (screw speed and configuration), and CR particle size distributions by characterizing the mechanical performance of TPE dynamic vulcanizates (TDVs) through tensile and hardness testing and their morphology by SEM micrographs on the fracture surfaces of tensile specimens [1]. Balbay [73] examined the effect of recycled carbon-based material, obtained by chemical degradation of ELTs, on the mixture of tire tread (rubber composite) by comparing it with commercial CB-N550. Rheometeric properties, mechanical properties, hardness, morphology, functional structures, thermal degradation of the rubber composites prepared were determined. Zedler et al. [74] applied trans-polyoctenamer rubber, a commercially available waste rubber modifier, to GTR. They investigated the influence of various additives (sulfur, N-cyclohexyl-2-benzothiazole sulfenamide, dibenzothiazole disulfide and di-(2-ethyl)hexylphosphorylpolysulfide on curing characteristics, mechanical, thermal, and acoustic properties, and morphology of modified GTR for possible reclaim and crosslinking between applied components.

Zedler et al. [75] reclaimed and modified GTR with 10 phr of ethylene-vinyl acetate copolymer by low-temperature extrusion. They investigated processing and mechanical properties, Volatile organic compound emission, and recycling, and compared their efficiency with trans-polyoctenamer, an additive commercially dedicated to waste rubber recycling. Fazli and Rodrigue [76] used GTR as a partial replacement to virgin rubber. They reviewed possible physical and chemical surface treatments to improve GTR adhesion and interaction with different matrices, including thermomechanical, microwave, ultrasonic, and thermochemical rubber regeneration producing regenerated tire rubber (RTR). They discussed the effect of GTR/RTR particle size, concentration, and crosslinking on curing, rheological, mechanical, aging, thermal, dynamic mechanical and swelling of rubber compounds.

4. Applications of Devulcanized Rubber

The most established way to devulcanized and revulcanized elastomers is producing polymeric blends, meaning physical mixing devulcanizated rubber with a polymer or more. Polymer blending or compounding aims to improve the properties of devulcazinates to neat polymers, i.e., obtain materials with improved physical and mechanical properties while decreasing the costs of compounds and improving processing [1].

Virgin and devulcanized rubbers can become part of many different compounds. These elastomer-based blends divide into two types: i. Blends composed of two or more elastomers and ii. Blends made of thermoplastic elastomers or TPEs and elastomeric phases. The compounds are TPEs when the main phase is elastomeric and toughened plastics when the main phase is thermoplastic. Still, revulcanizing a devulcanized rubber is more complex than vulcanizing virgin rubber, because devulcanization chemically changes the nature of rubber in ways not always predictable.

4.1. Blends Based on NR

For devulcanized rubber, the greatest attention goes to NR-based blends and composites, as in mechanically devulcanized CB-filled NR by shearing through a two-roll mill. To simulate recycling WTR parts, devulcanized samples aged at 70 and 100 C for days mixed in virgin NR at 85/15, 75/25, and 60/40, with various amounts of fillers added during blending. Blend properties adjusted by adding fillers, proving mechanical devulcanization and compound optimization in recycling NR. Results compared to data reported on blends incorporating up to 50 phr of mechanically devulcanized and non-devulcanized GTR in NR [1].

Further work compared different devulcanization strategies on the properties of final blends, since each devulcanization process uniquely affects the structure of rubber thus the properties of final blends. Comparing GTR $scCO_2$, ultrasonic, and biological devulcanization and blending the devulcanizates in NR at 10 phr concentration, ultrasonic and $scCO_2$ yielded higher devulcanization, while biological treatment, although more selective, was limited to the surfaces. The mechanical properties of blends containing biologically dGTR compared to those containing non-devulcanized GTR, while GTR from ultrasonic treatment showed the greatest improvement in mechanical properties, up to a certain ultrasonic amplitude.

4.2. Blends Based on Synthetic Rubbers

Valentini et al. [1] compounded various amounts of devulcanized and non-devulcanized recycled rubber from truck tires with virgin EPDM rubber, and resulting compounds expanded with azodicarbonamide. Devulcanized particles encapsulated better within EPDM matrices to show interfacial adhesion, probably due to revulcanization in which free crosslinking sites formed further linkages with EPDM. Tensile and impact behaviors of expanded EPDM/recycled rubber blends highlighted improvement of total absorbed energy, impact strength, and elongation at break compared to virgin EPDM for all investigated compositions, especially with a devulcanized rubber content of 30 wt%. The preparation of expanded EPDM containing considerable amounts of devulcanized rubber still proved plausible to reduce the costs and improve the properties and environmental sustainability of rubber goods [1].

Other researchers reported devulcanization of GTR and SBR crumbs using microwave processes prior to mixing with virgin SBR. Devulcanization significantly increased the properties of blends compared to blends with non-devulcanized rubber powder. They measured strains at break of 445% on composites with dGTR and only 217% on composites with untreated GTR. This relates to improved interfacial interactions between treated GTR and the surrounding matrices, as evidenced by SEM [1]. Further researchers compared low-temperature mechano-chemical devulcanization (LTMD) and traditional high-temperature atmospheric devulcanization to reclaim waste rubber powders mixed with additives, then revulcanized the reclaimed rubber after blending with all-terrain vehicle tread rubbers. They noticed that not only LTMD yielded a higher devulcanization fraction, but the blends devulcanized also reached higher mechanical properties even without additional additives.

4.3. Blends with Thermoplastics

Devulcanized rubber allows for full-elastomeric blends and rubber/TPE compounds. It considered PP, high-density polyethylene (HDPE), copolyester, and PS. Mechanical-chemical devulcanization of WTR powder mixed with PP in different proportions indicated decrease in hardness of the devulcanized rubber fraction and increase with radiation up to 50 kGy especially at higher devulcanized rubber fractions. Tensile strength also increased with radiation but was strongly impaired when incorporating devulcanized rubber. Strain at break increased with devulcanized rubber loadings of up to 75 wt% but decreased with radiation doses. Devulcanized rubber proved good a filler to tune PP mechanical properties.

Researchers prepared GTR/HDPE blends by combining GTR chemical surface devulcanization with tetraethylene pentamine (TEPA) and in-situ grafting of HDPE by styrene and glycidyl methacrylate. This strongly improved blend compatibility and mechanical properties, and the properties adjusted with the initiator/grafting monomer ratio. Moreover, dGTR/HDPE blends proved stable for processing and reprocessing [1].

Other researchers incorporated devulcanized ruber in a brittle matrix such as PS. Valentini et al. [1] melt-compounded and compression-molded devulcanized and non-devulcanized rubber from waste truck tires with PS. Devulcanized rubber domains were smaller and better dispersed in surrounding matrix, and exhibited improved interfacial interaction compared to non-devulcanized rubber. However, surface hardness, tensile modulus, and stress at break dropped with limited compatibility between PS and rubber particles in relatively large rubber domains. High rubber fractions showed increase ductility, enhanced tensile strain at break, and resistance to impact.

Hittini et al. [1] used devulcanized rubber powder from waste tires as a filler for PS thermal insulators. Devulcanized rubber was added to PS in proportions up to 50 wt% using a melt extruder and the mixes resulting were hot pressed. Composites with less than 40 wt% devulcanized rubber exhibited superior properties, with thermal conductivity ranging from 0.050 to 0.071 W/mK, density from 463 to 482 kg/m^3, compressive strength from 11.7 to 7.5 MPa, and flexural resistance from 40.4 to 19.3 MPa. The characteristics further increased with devulcanized rubber alkaline treatment, which increased the interfacial adhesion between devulcanized rubber and PS.

4.4. Industrial Application

Devulcanization has not yet achieved full circular economy implementation (meaning, making new tires from rubber recouped from used tires), but this can change as increased engineering products develop and prove out. Thermo-chemical breakdown of a U.S. truck GTR a local tire processor supplied completed at Windsor’s laboratory, on a small scale. Super critical carbon dioxide (scCO_2) accelerated the devulcanization, in a laboratory autoclave, by providing the heat needed to activate an organic Diphenyldisulfide (DPDS) devulcanizing agent used. Several process equipment and the internal rheological laboratory allowed completion of these laboratory investigations. Rheology measurements ensured full devulcanization and guided the addition of revulcanization package on a laboratory two-roll mill (Figure 4).

4.5. Experimental Program

The program included:

i. Running rheological curves of the devulcanized rubber;

Fig 4. Devulcanized waste rubber compounding

i. Calculating the required curing agent-accelerator using the composition mixture details;

ii. Molding slabs and buttons of the devulcanized rubber;

iii. Measuring tensile modulus on various samples of the devulcanized rubber;

iv. Making a small basic product, replacing the virgin polymer by increments of 10%;

v. Preparing a formula using the devulcanized rubber as 100% of the polymer to achieve the properties sought for an industrial application, and

vi. Making a new product, a front parking block for passenger vehicles, using the new formulation and testing it

Table II presents the composition of rubber used in this investigation, with percentages, measured, and standard deviations, calculated based on six repeated measurements that completed successfully.

4.6. Experimental Results

Table III presents results of tests on rubber obtained by laboratory thermo-chemical devulcanization at Windsor's laboratory.

Table II. Truck GTR Composition for Devulcanization

Materials	Pecentage (%)	Range
Natural Rubber (NR)	36	+/- 2.5
Synthetic Rubber (SBR)	21	+/- 2.0
Polymer Content	57	+/- 3.0
Carbon Black	26	+/- 2.5
Ash	8	+/- 1.0
Acetone	9	+/- 1.5

The table reports average physical and rheological properties from several samples (six to 12) tested. It also reports standard deviations calculated from the multiple tests completed.

Table III. Devulcanized GTR Characteristics

Property	Specifications	Value	Range
Tensile Strength	ASTM D412	10 MPa	+/-1.5
Elongation	ASTM D412	250%	+/-50.0
Mooney Viscosity	ASTM D1646	45	+/-15.0
Density	ASTM D297	1.13	+/-0.03
Hardness	ASTM D2240	55 Shore A	+/-5.0

The summary and key findings on samples, in light of experiences on natural and synthetic, at Windsor's laboratory and at some of its customers interested in recycling post-industrial scrap rubbers, indicate that:

i. Devulcanized rubbers are not compounds, but replacements for polymer contents of the ingredient list of products to make;

ii. From understanding Organic Chemistry of the vulcanization process of natural and synthetic rubbers, the devulcanized rubbers do not have the molecular behaviour expected from virgin polymer structures, and

iii. The molecular structure of the virgin polymer equates to a predictable Glass Transition to consider for developing products requiring safety (example; automotive tires).

Still, samples from the devulcanized rubber using thermo-chemical devulcanization with $scCO_2$ and DPDS in an autoclave tested as a compound, with the data reported in Table III - no addition of any virgin rubber. The study aimed at making a large static product, a front passenger vehicle parking block with 100% devulcanized rubber from scrap truck tires for a maximum sizable usage of waste rubber.

4.7. Application Development

Certainly devulcanization has not yet achieved full circular economy implementation (meaning, making new tires from rubber recouped from used tires), but this can change as increased engineering products, similarly to what is reported in this paper, get developed and proved out. In 2004, a global evaluation conducted on the utilization of devulcanization technology for tire recycling in Europe. Devulcanization appeared to exist only for research and development purposes at a scale of no more than 45 kg/h and still, on a sporadic generation. At that time, researchers stated, "devulcanization faces an uphill struggle to be competitive with virgin rubber".

The observed barriers were a lack of reliable data on devulcanization, no clear standards, short-term calls, and price-driven markets. Since, however, devulcanization continued to develop to find ultimately a place on industrial scales. In particular, Windsor's laboratory is using three decades of rubber product development to engineer novel rubber products from recycled rubbers.

Figure 5 is the first product the laboratory developed for molding and is gearing to mass-produce. The laboratory is targeting large volume applications (for sizeable fits to recycled rubbers) of limited liabilities, to start. The system breakdown of the first product on Figure 6 shows a bulky recycled rubber block for passenger vehicle parking, on which attach three textured patches to reflect light for viability in poor lighting or weather conditions, and an installment kit made of plastic dowels and galvanized steel screws and washers to protect with plastic caps. The assembly installed, since, on several parking lots in Ontario (example on Figure 7).

Current parking products are mostly made of concrete. Concrete parking blocks for passenger vehicles on the market crack with weather changes (cold to hot) and break at impacts. They are heavy, raising difficulties to bring to a job site and to install, requiring heavy machinery, and still, damage softer asphalt underneath. They change color under the elements (sunlight, rain, and snow) and build mould (fungi). They are unpleasant aesthetically, and become an eyesore years in service. They are not recyclable, and their generation utilizes many processes of mixing, pouring, curing, each, with substantial consumptions of energy and disposals of greenhouse gases to the atmosphere. They cause hazards of pedestrians and vehicle owners parking vehicles, tripping to fall on hard surfaces. Still, most dramatically, they scratch vehicle fenders and underneath, especially those with low profiles and chassis closer to the ground for better handling and maintenance on roads.

The recycled rubber, front parking block, for passenger vehicles, Windsor's laboratory developed and is promoting, circumvent all the issues reported above, and offer:

Environmental Responsibility. Using scrap tires to make parts re-uses masses of rubber that would otherwise go to waste every year. Recycled rubber blocks eliminate 200 scrap tires in a 60-slot parking lot. It does not take a large project to make a sizable dent in our huge ELT waste problem. Algeria discards about 0.1 mt (metric tonnes) of scrap tires each year. Three quarters of these can divert away from incineration and landfills, into similar product development endeavours.

Weather Resistance. Cement parking blocks crack, break, change colour with the elements, and build mould. They are heavy to move around and damage the pavements below. Rubber blocks eliminate these issues and avoid scratching the underneath of fenders of vehicles when parking close to a block.

Greater Longevity. Rubber blocks resist the weather compared to cement blocks. Water in cracks can freeze to break the cement blocks.

Improved Economics. Cement blocks are steel-reinforced and require many moulds as curing takes around 24 hours. Conversely, one single mould makes several rubber blocks per hour.

Fig 5. Passenger vehicle parking block made from WRT

Increased Safety. Markings stand out on rubber blocks, making parking at night and in poor weather conditions easier. Rubber absorbs hits, cushions slips and falls, and does not damage vehicles. It is also easy to install and remove, and is light to bring in to and take away from job sites.

Windsor's laboratory developed technologies to i. Recover rubber from products containing rubbers, like tires, ii. Devulcanize the recovered rubbers, iii. Compound the devulcanized rubbers, and iv. Manufacture with recycled (devulcanized and compounded) rubbers, materials and products. Currently, the laboratory offers two lines of products:

Recycled Rubber: This can be recycled SBR pellets to replace SBS polymer in asphalt to rubberize roads, parking lots, driveways, etc. Windsor's laboratory also offers devulcanized rubbers for mixing, to reduce the cost of new compounds.

Recycled Rubber Products: The laboratory is moulding under contract a small solid idler for the Mining Industry for a heavy machinery manufacturer in the GTA (Greater Toronto Area). It is also launching its first product, namely a front parking block for passenger vehicles.

The laboratory offers services in terms of receiving and treating post-industrial polymeric wastes involving rubbers as well. One such application is temporary seals around vehicle doors from General Motors at the GM facility in Warren, Michigan. Talks are also underway with various other vehicle Original Equipment Manufacturers (OEMs) and different level tire suppliers.

Other products are being developed under the EcoCa™ brand as well, and include various markets and industrial sectors spanning automotive, oil and gas, leisure, etc. Some of the applications that we are present in are:

i. Truck front parking blocks;

ii. Bus front parking blocks;

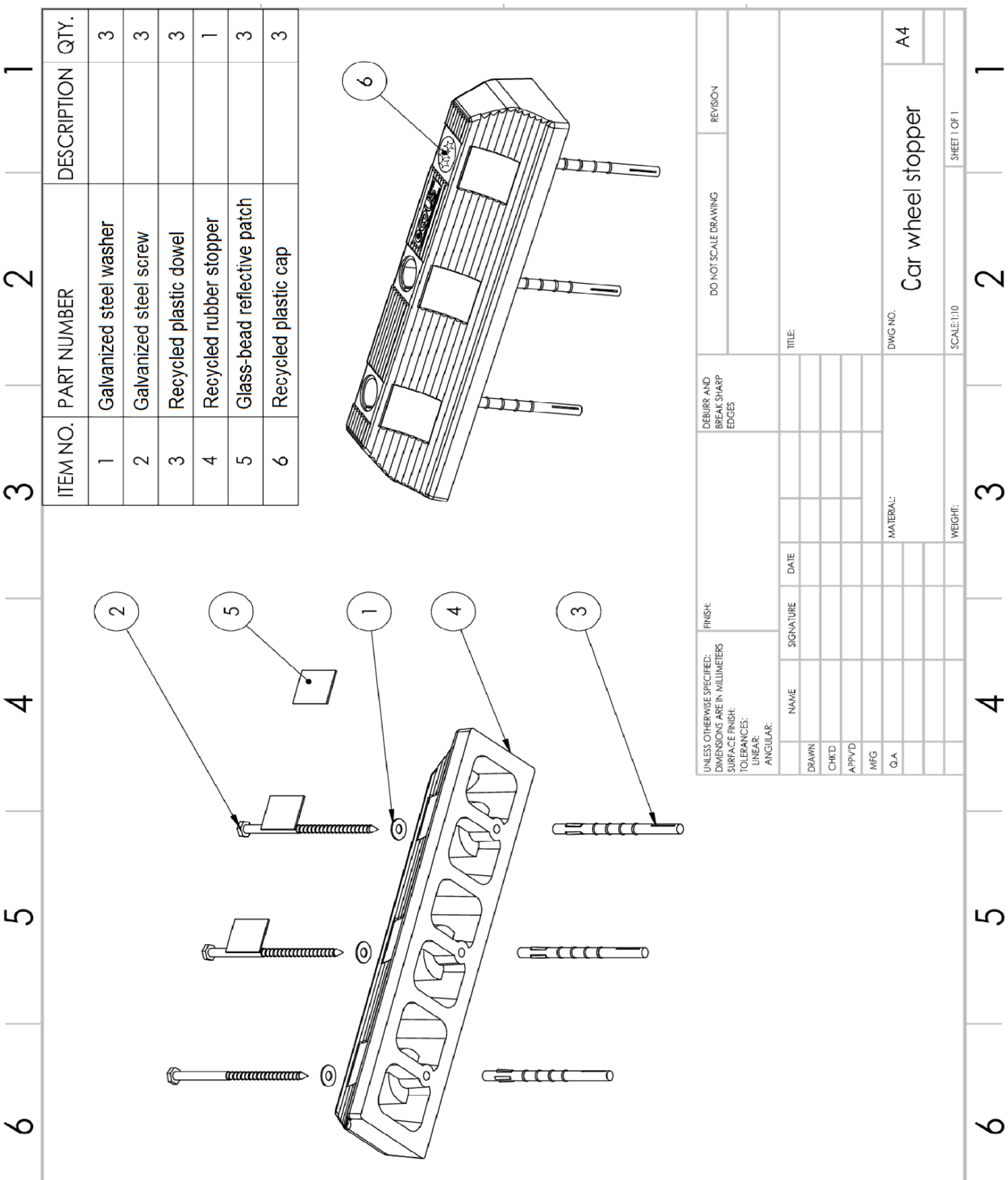

Fig 6. Passenger vehicle parking block made from WRT

i. Lane dividers (between bikes and vehicles);

ii. Barrier-post joints along highways;

iii. Sleeve covers to bollards;

iv. Entrance and antifatigue pads;

v. Fenders for boats, and

vi. Fenders for marina and port docks

Additionally, Windsor's laboratory is open to engineering applications for customers. Developing engineered (functional) products from rubber recouped from scrap tires involves three main technical ingredients: i. Rubber chemistry (in particular, the devulcanization of vulcanized rubbers), ii. Rubber product design and engineering (based on computer-aided design, CAD, and simulations based on finite element analysis, FEA, and computational fluid dynamics, CFD), and iii. Rubber manufacturing with rheology and tooling design and machine processing (manufacturing), to optimize on a computer then make.

The laboratory team in Windsor and extended team in various geographies are made of veterans in the Rubber Industry and each member offers advanced academic credentials and decades of industrial experiences in material development and characterization, rubber chemistry, product design and optimization, manufacturing processing and fine-tuning, tooling, prototyping, and testing.

Fig 7. Devulcanization processes reported in the literature

1. Conclusions

With the continually-increasing development, production, consumption, and disposal rates of scrap rubbers and rubber products as industrial and post-usage wastes, adequate valorization of these valuable materials is urgent. The technical analyses of work completed, the summary of which making for this publication, lead to the following observations:

i. As landfilling and open burning (still practiced in many regions and in the open in some poorer geographies) are unsustainable and polluting, downsizing waste rubber to pellets and powder continues even if reaching saturation. Crumb rubber adds to cementitious and rubber-based blends to produce rubberized asphalt and concrete, playground surfaces, running tracks, etc. However, this approach fails to find fits for rubber wastes generated yearly, and the scarce adhesions between rubber particles and surrounding polymer matrices strongly limit the amount of rubber waste to incorporate in other materials.

ii. The most suitable approach, perceived today, to sustainably recycle rubbers and products container rubbers (like tires) is devulcanization, an undertaking that selectively breaks the S-S and C-S bonds in rubber networks, leaving the backbones intact, to recover a rubber similar to the rubber before vulcanization, to compound and manufacture into highly-engineered products.

iii. Devulcanization involves mechanical, thermal, thermomechanical, chemical, microwave, ultrasonic technics, or biological agents, solo or combined. All methods present advantages and shortcomings, calling for combinations to enhance devulcanization outcomes, avoid backbone degradation, and optimize the characteristics of devulcanizates. Properly selecting the devulcanizing agents and operational conditions, like temperature, pressure, time, and shear rate, are important.

iv. Devulcanized post-industrial and post-consumer rubber wastes can partially replace virgin rubbers, thereby saving virgin feedstock, reducing costs, and saving natural resources. Devulcanized rubbers can also mix with different elastomers and thermoplastics, into blends with sought characteristics. Nonetheless, rubber devulcanization should foresee its revulcanization and the role of additives and fillers in manufacturing.

Many challenges remain open to implement rubber sustainability through effective rubber devulcanization then the development of sustainable markets for devulcanizated and revulcanizated rubbers ad rubber products. This relies on in-depth developments and precise correlation among physical and chemical characteristics of vulcanizates, devulcanization processing, and the resulting devulcanizates, along with manufacturing with devulcanized rubbers.

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by the author or related to Windsor Industrial Development Laboratory.

Conflict of Interest

The author declares that he has no conflict of interest related to the work by Windsor Industrial Development Laboratory reported herein.

Data Availability Statement

Not applicable.

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