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Production of Automotive Primer Using Nano/Microparticles Obtained from Expanded Polystyrene Packaging Waste

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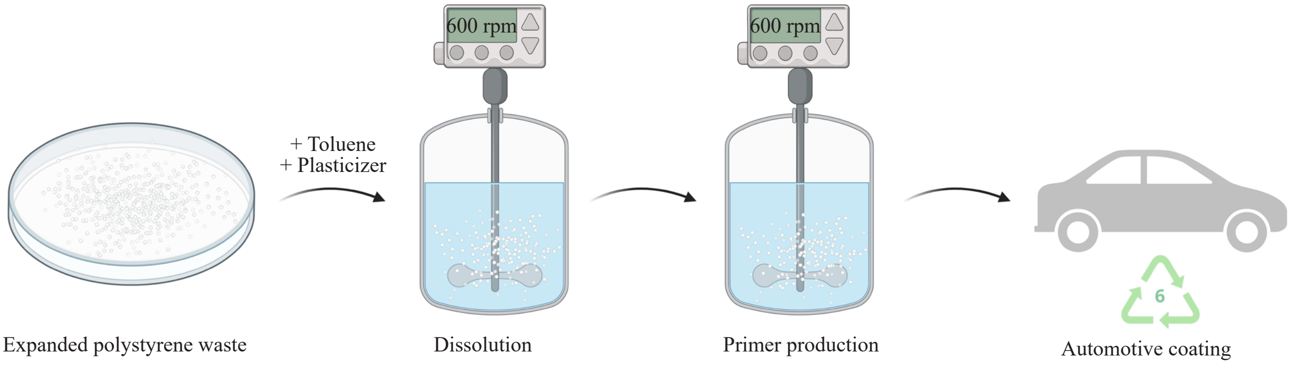
Graphical Abstract:



Abstract: Polystyrene nano/microparticles obtained from post-consumer expanded polystyrene waste (WPS) were dissolved in toluene and incorporated into a commercial-grade automotive primer. The use of WPS situates the developed product within current sustainability and circular economy concepts. The main objective of this study was to develop an automotive primer containing 2 and 4 wt.% of nano/microparticles. For this purpose, WPS particles were dissolved in toluene and added to the primer, which was formulated with various compatibilizers and plasticizers. The resulting P03 primer exhibited chemical, rheological, mechanical, and morphological properties comparable to those of standard commercial primers, demonstrating that the addition of WPS-derived nano/microparticles does not compromise performance. This approach provides a promising recycling route for WPS, avoids secondary waste generation, and aligns with the principles of sustainable development and the circular economy.

***Keywords*:** plastic waste, expanded polystyrene, dissolution, recycling, primer automotive refinishing

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1. Introduction

Polymers are versatile and have low production costs due to their extensive use in the modern lifestyle.[1-5] Global plastics sales reached approximately 437 Million Tons (Mt) in 2022, reflecting the continued increase in demand and worldwide dependence on these materials.[6] Their immense use makes recycling an indispensable measure to avoid the depletion of fossil fuel reserves.[7-10]

Expanded Polystyrene (EPS) is a homopolymer obtained from the polymerization of styrene in the presence of an expanding agent.[11-14] The material is not abrasive and has low density and good resistance to mechanical vibration, humidity, water, and mold. It is thermal and acoustic insulation, a set of properties that make EPS a versatile material with several applications, such as food and general use packaging, civil construction, the automotive sector, and sporting goods. The low density of EPS caused by air inside the material brings advantages, making logistics challenging to dispose of after use. Not only does transportation become more expensive, but so does disposal in landfills; besides that, improper disposal causes several environmental problems since the waste has a long degradation time.[14-21]

Among the alternatives, the dissolution technique is effective as an intermediate in recycling, generating a solution with the residue dispersed in the solvent,[11,22-25] enabling the production of other products, and obtaining pellets for general use or nanoparticles. The dissolution technique is the solubilization of a single polymer or a solvent mixture. It has advantages such as the preservation of polymer chains, reduction of the volume of waste, and costs associated with transportation if done at the source of production.[23,26,27] In addition to enabling the production of pellets for general use, the dissolution technique also facilitates the production of nano/microparticles, which adds significant value to recycled materials.

In this context, expanded polystyrene waste (WPS) nano/microparticles can be obtained through the solvent/antisolvent technique, a variation of the dissolution process that allows the transformation of polymer waste into high-value materials. The method consists of dissolving WPS in ethyl acetate, a renewable and environmentally friendly solvent, followed by nanoprecipitation in ethanol, which acts as an antisolvent, under vigorous stirring. The formation and size of the particles are influenced by factors such as the concentration of polystyrene, stirring intensity, and the ratio between solvent and antisolvent. The resulting nano/microparticles exhibit spherical morphology, high chemical and thermal stability, and are free of residual solvents. The process is notable for its sustainability, due to the use of green solvents and the possibility of solvent recovery by distillation, as well as for its economic and technical feasibility, representing a promising alternative for producing functional materials from plastic waste. These materials have potential applications in areas such as membranes, drug delivery systems, fuel additives, paints, and coatings.[23]

The paint and coatings sector is constantly evolving, driven by advances in product quality, cost competitiveness, and the adoption of increasingly efficient and sustainable processes. The technologies applied in automotive coatings and surface finishing represent the cutting edge of innovation in this field, offering durable and aesthetically enhanced solutions that combine high process efficiency with compliance with environmental requirements.[28-30] The primer, a preparatory layer applied to a vehicle's surface before the final paint application, is a key product within the automotive refinishing market. It is composed of resins and binders, pigments and fillers, solvents, and various additives. Serving as a foundational layer, the primer is applied before the basecoat, which provides color, and the clearcoat, which delivers gloss and protection. It is designed to ensure excellent adhesion of subsequent layers, level surface imperfections, and offer strong resistance to chipping and mechanical damage. The product can be produced in solvent, water-based, or powder form, the first being the most used.[31-33] Given this scenario, the incorporation of recycled nano/microparticles into coatings, such as automotive primers, emerges as a sustainable strategy that aligns technological innovation with environmental responsibility.

This study aimed to obtain an automotive primer by incorporating nano/microparticles of expanded polystyrene waste, valuing the residue, and minimizing the use of virgin raw materials. For this, an automotive primer with part of its commercial resin was prepared and replaced by one based on WPS. It was later compared with the original product to maintain a product with the minimum requirements and the best possible replacement. The characterization of the primers was carried out through measurements of viscosity, density, König hardness, resistance to solvents, crack resistance, and morphological surface. This work aims to offer new recycling perspectives to WPS through new processes and standard characterization techniques, which can provide basic information for future considerations about their uses in large-scale recycling processing.

2. Materials and methods

Anjo Tintas Ltda (Santa Catarina, Brazil) supplied the toluene and the automotive primer. Resume Recycling and Trade Ltda (Santa Catarina, Brazil) supplied the WPS (Mw: 188 kDa).

The nano/microparticle samples were provided by the Laboratory for Control and Polymerization Processes at the Federal University of Santa Catarina (LCP/UFSC). The WPS recycling process was based on its dissolution in a solvent, followed by precipitation using an antisolvent.[23] Post-consumer WPS residues were previously cleaned, ground, and dried before processing. The material was then dissolved in ethyl acetate, and the titration of this solution was performed in ethanol, which was used as the antisolvent. After polymer precipitation, the solvent was evaporated, resulting in the formation of WPS particles. The particles were subsequently analyzed for average particle size using Dynamic Light Scattering (DLS) with a Malvern Zetasizer Nanosizer. The samples were previously diluted in distilled water from 0.01% to 0.1% in order to obtain an ideal concentration for analysis and avoid multiple scatterings. The measurements were performed at a temperature of 25 °C, with a detection angle set at 173° (backscattering mode), suitable for colloidal suspension particles. The sample was analyzed in triplicate, with 20 runs per measurement, and the results were expressed as mean hydrodynamic diameter (mean z) and Polydispersity Index (PDI). The hydrodynamic diameter was 485.3 nm, and the PDI was 0.448, indicating a broad and polydisperse size distribution. The presence of particles across a wide range of sizes likely results from aggregation phenomena, sample heterogeneity, or a mixture of materials with distinct characteristic dimensions, as illustrated in Figure 1.

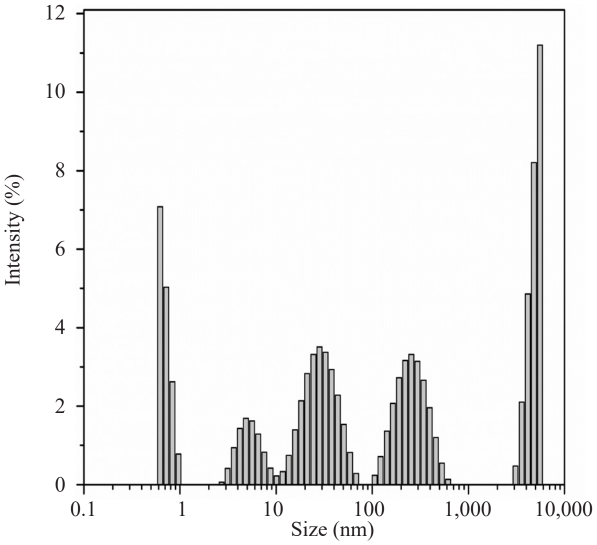

Figure 1. The particle distribution of WPS nano/microparticles used in the manufacture of automotive primers

A commercial primer was used in this study as a standard for comparison purposes. It consists of 22% by weight of resin, 48% by weight of mineral filler, 25% by weight of solvents, and 5% by weight of additives (Anjo Tintas, Brazil).

To prepare the primers for the study, the nanoparticle/microparticle was first gradually dissolved in toluene (RED number: 0.78) with the aid of a mechanical stirrer rotating at 200 to 600 rpm until a concentration of 4.1% by weight was reached. At the end of the dissolution of the WPS nanoparticle/microparticle, the plasticizer was added. Different plasticizers were used to evaluate their influence on the final product, namely Dibutyl Phthalate (DBP), Acetyl Tributyl Citrate (ATBC), Styrene-Ethylene-Butylene-Styrene (SEBS), and Dioctyl Adipate (DOA). Using an analytical balance and a mechanical stirrer, the primers for the study were prepared by replacing the original resin with resins containing 5% and 10% WPS in their composition. The compositions corresponding to the substitute fractions of the resin are shown in Table 1.

Table 1. Fractions of replacement of the original resin by WPS

Code	Plasticizer	Plasticizer wt.%	WPS wt.%	Toluene wt.%
P01	DBP	0.8	0.1	2.0
P02	ATBC	0.8	0.1	2.0
P03	SEBS	0.8	0.1	2.0
P04	DOA	0.8	0.1	2.0
P05	-	-	-	2.0
P06	DBP	1.6	0.2	4.0
P07	ATBC	1.6	0.2	4.0
P08	SEBS	1.6	0.2	4.0
P09	DOA	1.6	0.2	4.0
P10	-	-	-	4.1

WPS concentrations of 0.1 and 0.2 were selected because higher amounts could compromise the structural integrity of the matrix, leading to phase segregation, heterogeneities, or excessive softening. Therefore, the chosen range represents a safe window to enhance flexibility without negatively affecting the morphology or the mechanical properties of the material.

For the adhesion tests with tape and crack resistance in the conical mandrel, the one proposed by American Society for Testing and Materials (ASTM) D609[34] was followed to choose the specimens and cleaning. Steel sheets 200 mm (length) × 150 mm (width) × 0.8 mm (thickness) were cleaned with cotton cloths soaked in turpentine (Anjo Tintas, Brazil).

For the primer application, the recommendation by ASTM D823[35] was followed and air-pressurized handguns were used. With the tool pointed directly on one end of the plate, it was moved approximately 5 cm outward, and with the trigger pressed, the gun was moved perpendicularly towards the opposite end until it overtook it; the same procedure was repeated, inverting the direction of application. This process characterizes a coat. A second coat was applied after 10 minutes. An acrylic enamel was also used as a finish for the adhesion test with tape, following the same procedure for the primer.

2.1 *Characterization*

2.1.1 *Viscosity*

The viscosity of the samples was measured according to ASTM D1200[36] and Equation (1). The samples were placed into a stainless-steel Ford Cup viscometer n° 04, leveled, and with its hole blocked until the device overflowed. With the aid of a ruler, the excess was scraped, and then simultaneously, a timer was started, and the glass outlet was unobstructed.

$$v = 3.85 \times (t - 4.49) \tag{1}$$

Where v is the kinematic viscosity (mm^2 s^{-1}), t (s) is the flow time.

2.1.2 *Density*

The sample density was evaluated according to ASTM D1475.[37] With a precision analytical balance (model ATY-224, Shimadzu, Japan), the mass of a 50 g stainless steel pycnometer, previously dried, was verified. Subsequently, the device was filled to the edge with samples, capped, and the surplus cleaned. The mass of the pycnometer with the sample was measured, and the density (D_m) was determined according to Equation (2).

$$D_m = \frac{W - w}{V} \tag{2}$$

W is the pycnometer mass with the sample, w is the pycnometer mass, and V is the pycnometer volume.

2.1.3 *Rating adhesion by tape test*

According to ASTM D3359,[38] 5 horizontal cuts and 5 vertical cuts were made firmly and constantly creating a grid using a stylus blade. After the crashes were made, the detached chips were gently removed with a cotton cloth. A piece of 3M Scotch filament tape large enough to cover the entire grid was applied over the cuts and rubbed to ensure complete contact of the tape with the plate. After 90 seconds, the tape was pulled in a single, fast movement as close as possible to the plate, trying to form a 180°. The samples were compared with each other and the standard according to the percentage of coating removed.

2.1.4 *Mandrel bend test*

The resistance test was performed according to ASTM D522.[39] The bending was performed by a conical mandrel (TKB Instruments), which allows the metal plate to bend under the cone with a lever, resulting in a crack in the primer applied to the plate. With the aid of a ruler, the distance from the beginning of the plate, which corresponds to the beginning of the cone, was measured until the end of the coating break. The graph shows the percentage of elongation. Because the thickness of the primer layer directly interferes with the fold, the actual thickness of each specimen was measured with the aid of a micrometer. With the values obtained, the actual elongation was verified.

2.1.5 *Pendulum hardness test*

The hardness test was followed according to ASTM D4366.[40] With a 100 µm bar extender. Each primer was applied to glass plates previously cleaned with cotton cloth and turpentine. After 24 hours, the first measurements were made. The plates were placed in the pendulum hardness support (BYK-Gardner), and the balls were lowered until they were in contact with the primer; the pendulum was adjusted to 6° and then released. The device automatically counted each passage of the pendulum using a counter. The test was also carried out for 48 and 72 hours, and in triplicate at each moment.

2.1.6 *Solvent resistance*

For the solvent resistance test, glass plates were cleaned with cotton cloth and ethanol, and then, with the aid of a 100 µm bar extender, the coatings were applied and allowed to dry for 24 hours. After that, a flannel pack was soaked with solvent and placed on the applied film; after 30 minutes of contact, it was removed to evaluate the possible changes in the visual aspect.

2.1.7 *SEM*

Scanning Electron Microscopy (SEM) (TM3030 Tabletop Microscope, Hitachi, UK) was used to evaluate the surface of the primer with the incorporation of WPS. For SEM investigations, the samples were coated with gold (Sputter Coater SCD 005, BAL-TEC, Liechtenstein) for electric conduction.

3. Results and discussion

3.1 *Characterization*

3.1.1 *Viscosity and density*

The results obtained for the viscosity and density tests are shown in Table 2. It is possible to observe that the standard has a lower viscosity than all the primers developed since, in this type of two-component system, the chain containing the isocyanate group is usually linked to closed chains, such as toluene diisocyanate, di-cyclohexylmethane diisocyanate, or tetramethyl xylene diisocyanate, presenting many free spaces and allowing a more significant movement of the molecules.

Table 2. Results of viscosity and density for the primer standard and primers with WPS incorporated

Primer	Viscosity ($mm^2 \cdot s^{-1}$)	Density ($g \cdot cm^{-3}$)
Standard	200.08	1.508
P01	271.52	1.482
P02	271.62	1.481
P03	264.42	1.488
P04	272.54	1.492
P05	286.06	1.492
P06	328.81	1.499
P07	328.71	1.500
P08	297.91	1.503
P09	326.02	1.502
P10	342.00	1.497

As expected, primers from P06 to P10 have higher viscosity than their lower-concentration counterparts. This is due to the polystyrene chain's more excellent replacement of the isocyanate groups, which have higher rigidity and less flexibility. It was noticed that the primers P03 and P08 presented a lower viscosity than the others that were developed. The chosen plasticizer has a styrene molecule at each end of its structure, connecting the polystyrene chains, increasing free spaces and flexibility.[41-43] A high viscosity avoids runoff and problems with low thickness after application; however, care must be taken when applying with pressurized handguns, as incompatible pressure can generate a boiling coating or the "orange peel" aspect, which was observed in all specimens coated with the developed primers.[44]

As expected, there was no significant difference between the samples in the density values obtained. Still, there was only a slight increase as the substitution of commercial resin for WPS resin increased since the standard primer resin has a lower solids content.

3.1.2 *Rating adhesion by tape test*

Table 3. Results of the tape test and after enamel for the primer standard and primers with WPS incorporated

Primer	Tape	Enamel
Standard	4B	5B
P01	3B	5B
P02	3B	5B
P03	3B	5B
P04	3B	5B
P05	3B	5B
P06	2B	5B
P07	2B	5B
P08	2B	5B
P09	2B	5B
P10	2B	5B

The results of the adhesion tests are shown in Table 3, before and after the application of the finishing enamel; Figure 2 presents a representative sample for each result obtained. The incorporation of polystyrene resin (WPS) negatively affected the adhesion properties: the higher the proportion of residue in the primer, the greater the loss of adhesion observed in the metal sheets. Primers P01 to P05, with lower WPS content, presented classification grade 3B, while primers P06 to P10, with higher residue content, reached only grade 2B. The commercial standard primer exhibited minimal debonding, with a classification of 4B.

This reduction in adhesion is associated with the high Glass Transition Temperature (T_g) of polystyrene, which can cause vitrification of the polymer before the system is fully cured, limiting proper anchoring to the substrate. A decrease in molecular weight could slightly reduce T_g, while the addition of small molecules such as plasticizers may create free volume, reducing intermolecular interactions and allowing greater chain mobility. These mechanisms could contribute to improving adhesion.

During specimen preparation, all formulated primers showed a visual finish inferior to the standard, with an "orange peel" defect. This issue can be attributed to primer viscosity (proven to be higher than the standard) combined with inadequate application pressure. Such defects can be mitigated by optimizing dilution with appropriate solvents.

Applying the acrylic enamel as a finishing layer proved efficient in all coatings with WPS, yielding grade 5B for every specimen, without flaking. The acrylic enamel does not simply "mask" the loss of adhesion at the primer-substrate interface; instead, it provides additional adhesion mechanisms at the enamel-primer interface. Acrylic basecoats are typically formulated with acrylic resins that are compatible with polystyrene, enabling favorable intermolecular interactions and both physical and chemical anchoring.[45] This chemical and physical compatibility accounts for the restored performance in the multilayer system.

Overall, incorporation of WPS reduced direct primer-substrate adhesion (2B-3B vs. 4B for the standard). However, in the complete multilayer system (primer + enamel), adhesion performance was restored to 5B, due to synergistic bonding effects at the enamel-primer interface. The enamel, therefore, enhanced the overall system performance, but it did not eliminate the underlying primer-substrate adhesion limitation. This interface remains a critical aspect requiring further optimization (e.g., surface pretreatments, compatibilizer adjustment, or modified WPS content).

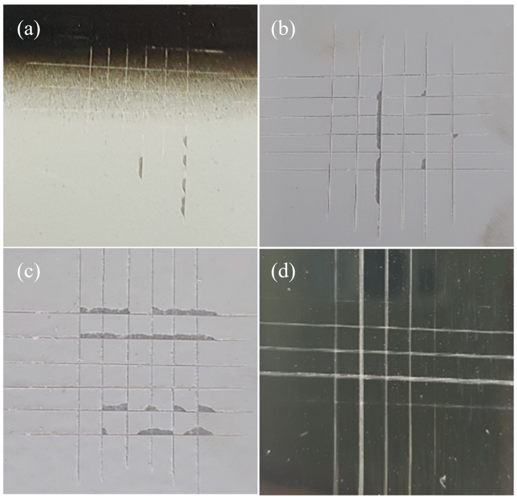

Figure 2. Tape adhesion test was applied with the primer (a) standard, (b) P03, (c) P08, and (d) P08 after the enamel was applied

3.1.3 *Mandrel bend test*

The values obtained in the mandrel bend tests were corrected to account for the thickness of each coating. The incorporation of expanded polystyrene in the primer negatively influenced flexibility and resistance under bending. In general, rupture extended beyond the limits of the specimens, demonstrating the lack of malleability of the material and making it difficult to clearly observe elastic deformation. Primer P03 achieved an elongation percentage closer to that of the standard primer, 5.11% (Figure 3a) and 7.74% (Figure 3b), respectively.

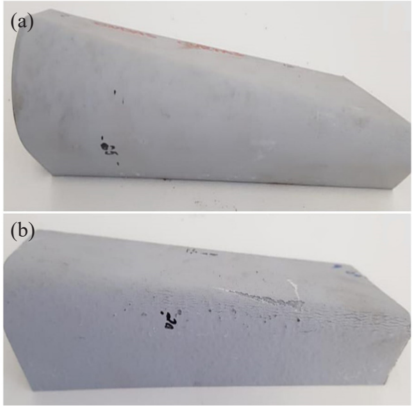

Figure 3. Image of the mandrel test applied with (a) standard and (b) primer P03

The improved performance of P03 is associated with the presence of the chosen elastomeric modifier, SEBS, which interacts with the polystyrene domains through its styrene blocks and increases chain mobility through its ethylene-butylene midblock. This effect reduces brittleness, decreases the effective Glass Transition Temperature (T_g), and allows greater elastic deformation. These macroscopic results are consistent with the known role of SEBS as a toughening agent in styrene systems, as widely reported in the literature.[46-48]

By contrast, primer P08, despite containing the same plasticizer, exhibited inferior performance. This suggests that either the higher content of WPS resins limited SEBS effectiveness or that the concentration of the modifier was insufficient, highlighting the importance of optimizing the formulation.

All specimens coated with the developed primers, including P03, still displayed pronounced cracks and coating peeling at the fold line. This observation corroborates the tape adhesion test results and reveals a deficiency in primer–substrate anchoring. While SEBS improved flexibility, it did not fully eliminate adhesion-related failure. This defect can be mitigated by employing appropriate plasticizers in optimized proportions, combined with surface pretreatments or compatibilizers, to strengthen primer-substrate adhesion.

Mandrel bending tests demonstrated that SEBS helps reduce brittleness and enhance the flexibility of primers containing WPS by increasing polymer chain mobility. Although Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC) analyses were not included in this study, they are planned for future work. These additional spectroscopic and thermal analyses will allow a more comprehensive understanding of the mechanisms underlying SEBS performance.

3.1.4 *Pendulum hardness test*

The film formation models determine that this is not an instantaneous process,[49] but the results of the hardness tests, as shown in Table 4, did not show a significant variation over time. Coatings with a high hardness have excellent resistance to damage, such as scratches and localized impacts. However, they tend to have less flexibility.[44]

The specimens coated with primers from P01 to P05 have a slight decrease in surface hardness compared to the standard. The reduction of the surface hardness is a sign of the weakening of the chemical bonds existing between the pigments and the polymeric matrix.[50] Still, reports in the literature showed higher values of hardness for coatings with lower thickness, as the proximity between the pigments and the polymer chains increases the forces between them.[51,52] Coatings containing a higher polystyrene content showed a higher surface hardness, an effect expected, corroborating the previous results.

One-way Analysis of Variance (ANOVA) revealed highly significant differences in König hardness across the primer formulations ($F(10, 22) = 476.50$, $p = 2.59 \times 10^{-23}$). The rejection of the null hypothesis provides statistical confirmation that the enhanced hardness properties observed in primers P06-P10 represent genuine performance improvements rather than random variation.

Table 4. Results of König hardness test for primer standard and primers with WPS incorporated

Primer	König hardness		
	24 h (cycles)	48 h (cycles)	72 h (cycles)
Standard	46	45	46
P01	43	44	44
P02	42	42	41
P03	44	44	44
P04	40	41	42
P05	42	44	43
P06	52	55	54
P07	54	53	53
P08	51	54	52
P09	54	55	54
P10	55	56	56

3.1.5 *Solvent resistance*

As expected, all solvents, Toluene, Ethyl Acetate, Butyl Acetate, and Ethylene Glycol, attacked the primer films. In specimens coated with primers from P01 to P05, as well as the standard, the appearance of boiling, tiny bubbles on the surface resulted from using a solvent with a very high evaporation rate, incompatible with the system. The result corroborates the one observed in the adhesion tests. It confirms a more accelerated cure of the polystyrene network to the polyurethane standard since the defect was not observed in the primers with a higher residue content.

3.1.6 *Scanning electron microscopy*

Since the previous results showed that the substitution of commercial resin for those developed based on WPS has a limit below 0.1 g/g of total resin, the micrographs performed were only for samples from P01 to P04 and the standard for comparison. The results of the tests showed compatibility between the components of the formulations, presenting a pseudo-homogeneous surface for the developed primers (Figure 4b) as well as the standard (Figure 4a). White dots in all samples indicate the presence of high mineral filler content, which is insoluble in the solvent.

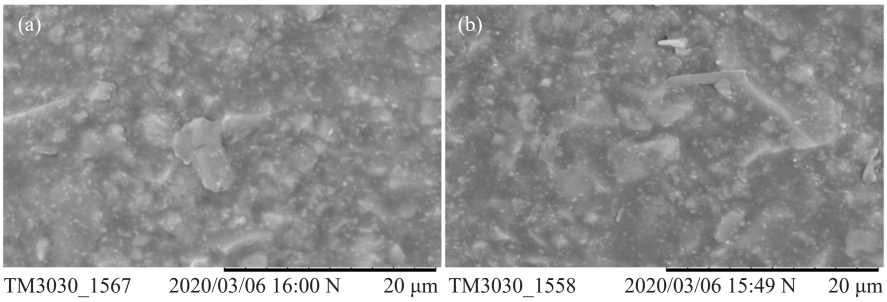

Figure 4. SEM of the specimens coating with the primer (a) standard and (b) P04

The specimens containing the primers with WPS showed micro-cracks along with the coating, which proves a deficiency of the used plasticizers.[41,53] The most significant cracks were present in the specimens of P01 (Figure 5a), exhibiting a crack length of 161.5 (± 2.1) µm, and P02 (Figure 5b), with a crack length of 125.0 (± 7.1) µm. The P03 primer (Figure 5c) applied showed more minor, narrow cracks, measured at a crack length of 92.5 (± 88.4) µm, reinforcing the previous results, while P04 showed no crack width observed.

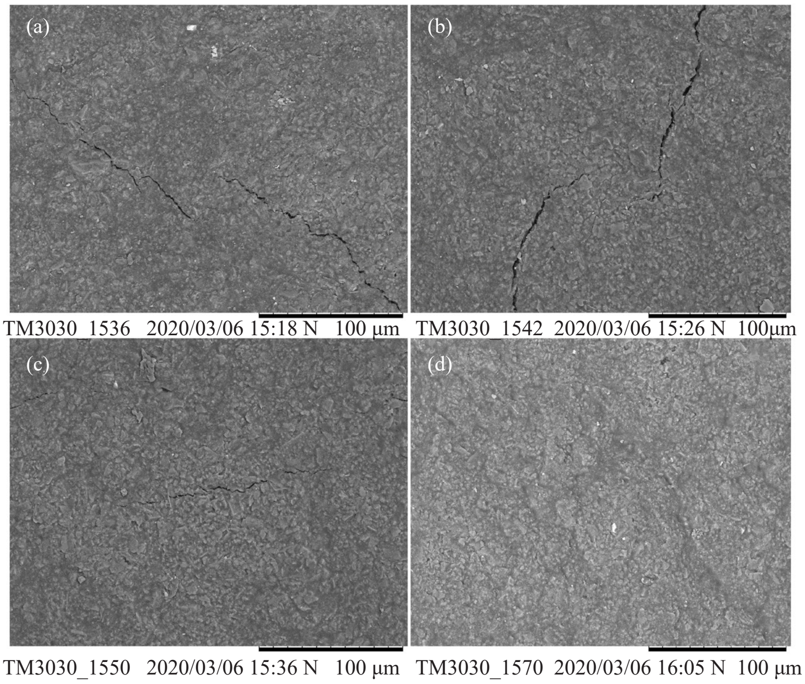

Figure 5. SEM of the specimens coating with the primer (a) P01, (b) P02, (c) P03, and (d) P04

Although the results demonstrated the technical feasibility of incorporating nano/microparticles obtained from WPS into automotive primers, this work presents some limitations that should be addressed in future research. First, the study was conducted on a bench scale, and the process has not yet been validated under pilot or industrial conditions, where mixing, curing, and solvent recovery dynamics may differ significantly. Second, only limited WPS concentrations (0.1-0.2 wt.% of total resin) were evaluated, as higher loadings led to defects such as loss of adhesion, brittleness, and phase segregation. This restricts the conclusions regarding the maximum feasible substitution content. The presence of microcracks and the "orange peel" effect also indicate the need for further optimization of plasticizer type and concentration, as well as application parameters. Finally, durability and weathering performance tests were not included, which limits the assessment of long-term behavior under real automotive conditions. Despite these limitations, the results provide a strong foundation for future studies aimed at scaling up the process, optimizing formulations, and validating performance under industrial environments.

4. Conclusions

The use of nano/microparticles of the expanded polystyrene resins in automotive primers proved promising, with the diffuse WPS between the polyurethane chains resulting in a homogeneous system. The choice of solvent is fundamental for the formation of the film and the solubility of the polymers. Toluene, which showed good dissolution of polystyrene, was also compatible with the rest of the system, forming a material as homogeneous as the commercial one. The detachment seen in the samples is because the WPS cures faster than the rest of the system. The problem can be overcome by evaluating the use of other solvents or a mixture with lighter substances, allowing the anchoring to the substrate. It was observed that the inherent hardness of the polystyrene propagated to the formed films, leaving them brittle, which shows that a higher substitution (0.1 wt.% of total resin) becomes unfeasible for the conditions of the used plasticizers. The plasticizer with the best results was SEBS, which forms bonds with the residue more quickly due to its styrene blocks, leaving the film malleable. Different concentrations of plasticizers can be tested to obtain a product more compatible with the market. Primers for industrial applications differ from those for automobiles because they require more durability in the weathering but less flexibility. The durability assessment may determine a different use from the one proposed in this work. Considering the chemical, thermal, mechanical, and structural properties of the material obtained, it is observed that it has appropriate characteristics that are equal to those of commercial grades. These characteristics are significant for recovering the material obtained in the recycled polymer market. There is no loss of chemical products added in the process, which saves the cost of treating secondary pollution. Two closed-loop processes were developed to recycle WPS and ensure its economic viability. This process is an investigative study at the bench scale. In future research, pilot-scale testing of these technologies will be carried out.

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Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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