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Materials and Clean Processes for Sustainable Energy and Environmental Applications

Development of a reliable and efficient combined grinding and separation process for PVC-coated fabric waste

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Abstract. This study aims to highlight a mechanical recycling process specifically tailored for PVC-coated fabric waste. While ample information exists on the recycling of pure PVC, a noticeable lack of information regarding PVC-coated fabrics has been identified in the literature.

Therefore, the primary challenge lies in accomplishing a thorough separation between PET fibers and PVC powder while simultaneously ensuring the optimal recovery of small PVC particles. This separation is crucial for maintaining the quality and purity of the resulting materials.

The effectiveness and efficiency of this process were evaluated through a comprehensive analysis that incorporated qualitative and quantitative studies.

For the qualitative study, an optical microscope was utilized to provide a rapid and non-destructive approach for evaluating the overall properties of the samples. This method provided valuable insights into the shapes, interactions, and cohesive bonds among the different components. In contrast, the quantitative study involved a chemical method that resulted in a destructive process. Using this technique, precise values were obtained, including the exact percentage of each constituent.

By integrating the findings from both qualitative and quantitative studies, informed decisions can be made regarding the selection and profitability of the method for achieving a complete separation between PVC powder and PET fibers.

The results obtained demonstrated a successful recovery rate of 83.92% for PVC compared with the initial amount. The remaining 16.07% of PVC was found to be either entangled with PET fibers or lost during the grinding process, particularly in the pulverizer machines.

Keywords. PVC-coated polyethylene terephthalate fabric, Chemical separation, Grinding process, Sieving, Characterization.

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1. Introduction

Artificial leather is a type of coated textile substrate created by applying a soft layer of polyurethane (PU) or polyvinyl chloride (PVC) onto a textile fabric base. Polyvinyl chloride (PVC) is widely used in the coating

industry because of its affordability, low density, excellent insulation properties, and high mechanical and thermal characteristics. However, PVC is inherently rigid and brittle, necessitating the use of plasticizers to enhance its flexibility when applied as a coating [1–4]. To formulate plasticized PVC, also known as plastisol, polyvinyl chloride powder (resin), plasticizers (such as diisononyl phthalate DINP),

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calcium carbonate filler, heat stabilizers, and pigments are combined [4,5]. The plasticizers and other additives are gradually added to the dry PVC resin and homogenized using a mechanical stirrer. Vacuum treatment is employed to eliminate air bubbles from the mixture, and when producing expanded layers, a puffing agent is incorporated along with the additives [6,7].

In applications where a thicker coating is required, PVC-coated polyethylene terephthalate (PET) fabric can be created by applying multiple successive coating layers [7–9]. This process typically involves three layers, as depicted in Figure 1: (1) a thin PVC superficial layer that serves for esthetic or technical enhancements, (2) a PVC expanded layer that functions as a sponge-like middle layer, and (3) a textile substrate layer composed of PET fabric, which imparts flexibility and strength to the material.

The production and consumption of PVC-coated fabrics generate significant amounts of waste. During the manufacturing process, scrap is discarded from the production lines. In industrial applications, most PVC-coated fabric-based parts are designed for single use, resulting in their disposal as waste once they become non-functional.

Similar to other composite materials, PVC-coated fabrics pose specific challenges in terms of waste disposal. The primary difficulty lies in the irreversibility of the PVC coating. Once a PVC part undergoes complete fusion and curing, it becomes impossible to recover the plastisol from the material [10].

Indeed, the issue of waste is exacerbated, particularly in the case of PVC-coated PET fabrics, as they possess inherent resistance to degradation, making it challenging for microorganisms to break them down [11,12]. When these PVC-coated fabrics are discarded in the environment, complete destruction may take several years. In addition, the presence of numerous additives in plastic-derived materials further contributes to aquatic pollution [13,14]. Therefore, the urgent need to identify an efficient approach for the treatment and recovery of PVC-coated fabrics has emerged as a critical environmental concern.

In the realm of waste management and recycling, PVC-coated fabric waste poses unique challenges that set it apart from pure PVC. These challenges stem from the distinct properties of PET fabric and plasticizer PVC, such as differing melting

temperatures, densities, and hardness. Therefore, conventional methods that effectively handle pure PVC cannot be directly applied to PVC-coated fabric waste. The mixing of these compounds, PET fabric and plasticizer PVC, during the recycling process compromises their original properties and practical functionality. Hence, the key hurdle in recycling PVC-coated fabric waste lies in effectively separating the PET fibers from the plasticizer PVC during the recycling process [14]. It is crucial to identify and implement the most appropriate combination of recycling and separation methods to address the environmental concerns resulting from the accumulation of PVC-coated fabric waste. By recognizing the optimal methods, a powerful strategy can be developed to mitigate the impact of PVC-coated fabric waste pollutants.

Extensive research has been conducted in the scientific literature to explore a wide range of recycling and separation methods specifically for pure PVC waste [11,13,15–33].

Each method possesses its own set of advantages and disadvantages, depending on the specific requirements of the user.

One commonly employed approach for disposing of PVC waste, especially when it contains a notable amount of impurities, is incineration [34]. Incineration, also referred to as quaternary recycling, remains a popular method for waste volume reduction and energy recovery [35,36]. However, incinerating PVC presents additional challenges because of its high chlorine content, which results in the release of substantial quantities of hydrochloric acid (HCl) during thermal decomposition [37,38]. The emission of hydrochloric acid gas contributes to the formation of acid rain [12,23,26–28].

An alternative recycling method for PVC-coated fabrics involves the chemical separation of the PET fabric from the plasticized PVC. This process begins by dissolving the PVC and then proceeds by filtering the fibers and distilling the solvent to reclaim the plasticized PVC [39]. Once successfully chemically isolated, PET fibers and PVC can be transformed into valuable raw materials suitable for diverse production processes [24,25,40,41].

The literature [10] emphasizes that PVC dissolves effectively in various solvents, some of which have known carcinogenic properties. However, tetrahydrofuran (THF) is regarded as the most suitable

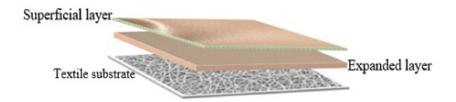


Figure 1. Different layers of PVC-coated fabrics.

because of its exceptional dissolution ratio, which can be achieved within just 10 min at 40 °C [33,42,43].

Chemical recycling through solvent processes is generally not widely accepted in the plastic industry for several reasons. These include the lengthy and costly nature of the process, the need for significant amounts of solvents, the potential alteration of the chemical structure and physical properties of the components, and its limited suitability for large quantities of waste [10]. To ensure the recovery of PVC with consistent quality and minimize the release of hazardous substances into the environment, it is crucial to explore appropriate methods. As a result, when the source of PVC waste is known, mechanical recycling is preferred [33,42,43]. Mechanical recycling is an efficient technique that upholds the inherent value of plastic by recycling waste materials into secondary raw materials while maintaining the material's chemical structure and composition intact [11,44]. For pure PVC, this approach provides accessible and cost-effective grinding facilities, environmentally friendly processes, and significant potential for profitability, particularly when compared with chemical or physical recycling methods.

However, despite the advantages of mechanical recycling, there is a current lack of comprehensive data available in the literature that can be readily adopted and implemented on a large scale within the PVC-coated fabrics recycling industry.

Therefore, an effective strategy for reducing the accumulation of environmental pollutants caused by PVC waste, particularly in industrial applications, would involve integrating recycling and PVC-coated fabric waste separation technologies.

This study aims to contribute to existing knowledge by developing a tailored process specifically for the recycling and separation of PVC-coated fabric waste. By combining insights from both qualitative and quantitative studies, informed decisions can be made regarding the selection and profitability of a

method that achieves complete separation between PVC powder and PET fibers. The qualitative study provides an overall understanding of the sample's characteristics, whereas the quantitative study offers precise data, enabling a more comprehensive evaluation of the separation method's effectiveness and efficiency.

Ultimately, the findings of this study will provide valuable insights into the recycling of PVC-coated fabric waste and contribute to the development of a cleaner and greener future.

2. Materials and methods

2.1. Raw materials

The PVC-coated fabric scraps used in this recycling study were generously provided by Plastics Company (Sayada, Monastir, Tunisia), a specialized manufacturer of transfer coatings. The relevant parameters of the raw material are presented in Table 1.

2.2. Plastic scrap grinder machine

The CMB Grinder machine was specifically designed for the primary grinding of thermosensitive PVC and other plastics to reduce them into smaller pieces. Equipped with powerful blades, this machine efficiently and swiftly grinds plastic scrap, facilitating the recycling process. The technical parameters employed for this machine are provided in Table 2.

2.3. SMF plastic pulverizer

The Carthage Plastic Company's pulverizer (Kaala Kbira, Soussa, Tunisia) was used in this study.

The plastic pulverizer machine is chiefly designed to grind PVC and fibers in micronized form.

This pulverizer uses a high-speed rotor. This rotor, which consists of several sections of disks and several

Table 1. Characteristics of PVC-coated fabric scraps

	Composition	Grammage g/m ²	Percentage
Coating layers	PVC	420	84%
Textile substrate	PET (Circular jersey, gauge 28)	80	16%

Table 2. Main technical parameters of the CMB grinder machine

MS 36		
per 2 rotors		
per 2 rotors		
kg/h		
_		

Table 3. Main technical parameters of the SMF plastic pulverizer

Model	SMF 600	
Milling fineness	20-120 (mesh)	
willing interiess	841–125 (µm)	
Rotor diameter	600 (mm)	
Capacity	70–350 kg/h	

blades, is responsible for grinding PVC waste into a fine powder. Its output can reach 70–350 kg/h (20–120 mesh). The technical parameters used are listed in Table 3.

In this process, the use of a vibrating sieve is crucial to ensure the desired output size. The vibrating sieve comprises two outlets: the fine powder outlet (7) and the coarse powder outlet (6) (refer to Figure 2). The fine particles that successfully pass through the sieve mesh are collected at the fine powder outlet (7). On the other hand, the coarse powders retained by the sieve are directed to the milling chamber for further grinding using the coarse powder outlet (6).

Regarding the PVC-coated fabric scraps, the fine powder particles are directed through the vibrating sieve with the assistance of air flow and collected in the product collector located at the fine powder outlet (7), designated as outlet P. However, the PET fibers, which are retained by the sieve, are vacuumed and subsequently recovered in a cloth bag connected to the coarse powder outlet (6), identified as outlet F (see Figure 3).

2.4. Optical microscopy

The morphology and structure of the expanded PVC layers were examined using a Leica DM 500 optical microscope. The microscope was equipped with various objectives connected to a color view camera and controlled by analysis software. The observations were conducted in the reflection mode at different magnifications of the objective.

To ensure accurate characterization of the particle images, it was necessary to utilize magnification and numerical aperture settings that provided sufficient resolution. The magnification factors employed ranged from $6.3 \times$ to $40 \times$, with the commonly used eyepieces typically set at $6.3 \times -25 \times$.

2.5. Test sieve shaker

Machine sieving is conducted by stacking the sieves in ascending order of aperture size and placing the powder on the top sieve (Figure 4).

A closed pan, acting as a receiver, is positioned at the bottom of the stack to collect the fines. A lid is placed at the top to prevent the loss of powder. This "nest" of sieves is vibrated until the residue on each sieve contains particles that can pass through the upper sieve but not through the lower sieve.

Typically, the results obtained from the sieving process are expressed as a cumulative percentage based on the nominal sieve aperture. For the analyses, RETSCH standard sieves were used.

2.6. Chemical separation

In the chemical separation procedure, tetrahydrofuran (THF) was used as the solvent to accurately isolate PET from PVC. A piece of the coated fabric was subjected to conditioning in an oven at 105 °C for over 5 h, followed by cooling to room temperature in a desiccator. The conditioned sample was then weighed and immersed in an Erlenmeyer flask containing THF solvent while being stirred.

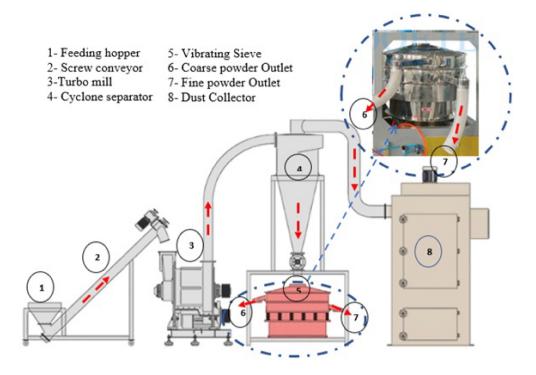


Figure 2. Plastic pulverizer.

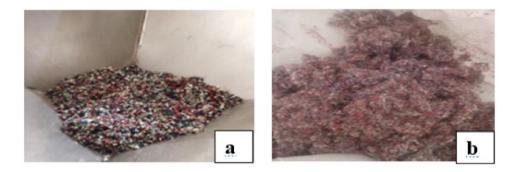


Figure 3. Pulverizer outlets: (a) recovered PVC powders (outlet P). (b) Recovered fibers (outlet F).

The solubility of PVC in THF depends on various factors such as the liquor ratio, temperature, and time.

To establish the effectiveness of the chemical method, several preliminary tests were conducted at different temperatures (20, 40, 60 °C) and durations (5–40 min). These tests aimed to validate the test protocol for chemical separation. It was observed that the solubility of PVC increased with higher temperatures, which were maintained using a silicone bath.

2.7. Distillation

In this study, distillation served as the second step in the separation process, primarily aimed at extracting the nonvolatile PVC from the THF solvent (Figure 5).

The homogeneous mixture comprising PVC and THF solvent was heated in a closed distillation flask. Because of its volatile nature, the THF solvent evaporated, forming a vapor. This vapor was subsequently cooled by passing it through the condenser,

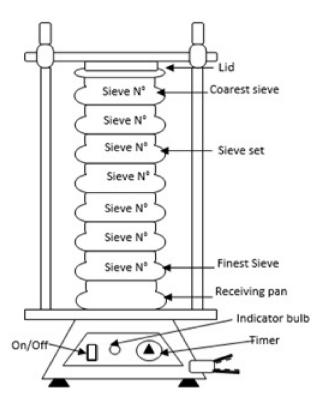


Figure 4. Machine sieving.

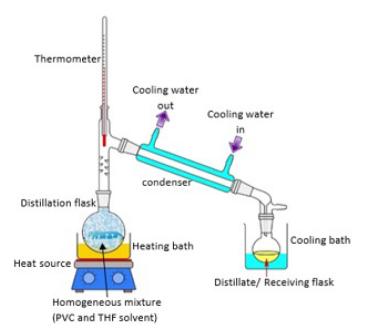


Figure 5. Distillation.

resulting in the condensation of pure THF solvent in a collection vessel. The recovered THF solvent can be reused for subsequent tests. The evaporation of the solvent facilitated the retrieval of the PVC component.

2.8. Sampling for analyses

The sampling procedure in this study aims to ensure that the collected sample accurately represents the complete physical, chemical, and morphological characteristics of the system under investigation. There are two key rules that should be followed during the sampling process:

- (1) Sampling of the powder should be conducted while it is in motion to prevent agglomerate formation. This ensures that the sample obtained is a true representation of the dispersed powder particles.
- (2) The entire sample stream should be collected over multiple short time increments, rather than selecting only a portion of the stream for the entire duration. This approach helps to capture variations in the powder composition and properties that may occur over time.

Therefore, in this study, the sampling procedure involved extracting small portions of powder from the bulk sample during the pulverization process, ensuring that the samples were taken from a moving powder stream. These samples were collected over several short time increments and later combined to form a representative sample, minimizing any potential sampling issues that may arise due to powder segregation.

3. Results and discussion

3.1. *Grinding process of PVC-coated fabric scraps*

The process of obtaining PVC powder from PVC-coated fabric scraps involves several stages of cutting and grinding. Initially, the waste PVC-coated PET fabric is cut into small pieces using a band saw, ensuring that their dimensions are suitable for the subsequent steps and equipment, with a maximum dimension of $1\ m^2$.

Subsequently, a first grinding process is conducted using a plastic waste grinder machine to crush the PVC-coated fabric component and

generate a bulk mixture, typically ranging between 10 and 15 mm in size. During this stage, the PVC remains combined with the PET knit, and they are not yet separated from each other.

Next, a second grinding process is employed using a pulverizer. This process further shreds the plasticized PVC component, resulting in the production of fine PVC powders that are effectively separated from the PET fabric (see Figure 3). An additional advantage of this grinding process, particularly with the involvement of a pulverizer, is its capability to not only grind the coated fabric scraps into PVC powder but also separate them from PET fibers. This characteristic provides an additional benefit to the overall recycling process. For visual representation, Figure 6 illustrates the considered grinding process map.

The following section presents a comprehensive qualitative and quantitative analysis conducted to investigate the behavior and effectiveness of the separation between PET fibers and PVC powder.

3.2. Qualitative and quantitative analyses

By integrating qualitative and quantitative approaches, a comprehensive understanding of the behavior and efficiency of the separation between PET fibers and PVC powder can be achieved. This synergy combines the advantages of rapid observations with the precision of constituent percentage measurements, resulting in a comprehensive analysis.

3.2.1. Qualitative analyses using microscopic observations

The qualitative study relies on the use of an optical microscope, which enables rapid and non-destructive assessment of the shapes and characteristics of the obtained components. This approach provides an insightful overview and facilitates a deeper understanding of the behavior exhibited by the samples, while ensuring that no damage is caused. Such observations play a crucial role in making initial decisions related to the process.

Separate quantities of PVC powder and PET fibers obtained from outlets (P and F) after pulverization were examined using image processing techniques. An optical microscope equipped with a high-resolution FEIQ250 Thermo-Fisher was used.

According to Figure 7, the PET fibers obtained from "outlet F" are observed to be covered with

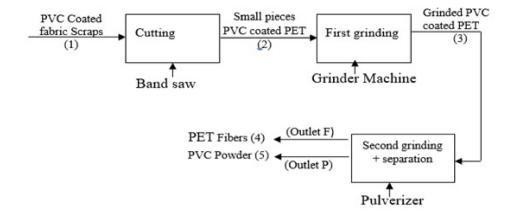


Figure 6. Grinding process map.

residual PVC. Microscopic observations reveal that these fibers are intricately entangled with the PVC particles, making their mechanical separation challenging.

Fibers were also observed in the case of output "P" (see Figure 8), indicating that the grinding process did not achieve complete separation of PVC from the PET fabric. To obtain more meaningful conclusions regarding the separation efficiency between PVC powder and PET fibers, a quantitative study was conducted.

3.2.2. Quantitative analyses using chemical separation

This quantitative study relies on a chemical method that involves the solubilization of PVC in THF. Despite the destructive and time-consuming nature of this method, it offers the advantage of yielding precise values, including the exact percentage of each constituent, after the grinding process.

To ensure reproducible results, it is essential to perform reliable sampling (see Section 2.8) for all analytical procedures prior to subjecting the samples to any analysis.

Chemical separation. The use of THF solvent revealed that the optimal performance for PVC removal was achieved at 40 °C within a 10-min timeframe, with a liquor ratio of 1:6 (Figure 9a). This outcome aligns with findings in the existing literature [45]. Following the treatment, the solution was filtered under an oil-pump vacuum, and the resulting insoluble PET fibers were thoroughly washed with water.

Subsequently, the fibers were dried at 105 °C for 24 h (Figure 9b).

In this study, distillation was employed as the second step of the separation process to extract the THF solvent from the nonvolatile PVC (Figure 5).

As depicted in Figure 10, the distillation process resulted in the recovery of highly pure plasticized PVC, which was devoid of any PET fibers or residual THF solvent. The recovered THF solvent can be reused in the next process cycle.

Chemical separation efficiency. Before quantifying the percentages of PET fibers and PVC powder, it is crucial to assess the effectiveness of the chemical separation method.

Microscopic examination (Figure 11) following THF treatment reveals that the knitted structure, composed of intermeshed loops, is visible. These results confirm that the THF solvent effectively dissolves the PVC without causing degradation of the PET fibers.

Furthermore, to validate the complete removal of the coated PVC and the absence of any residual THF solvent from the surfaces of the textile substrate, analysis using Fourier Transform Infrared Spectroscopy (FTIR) is recommended (Figure 12).

The band assignments for PET have already been reported in the literature [46–48] and are listed in Tables 4.

The observed results align with the spectrum of pure PET as reported by Ramesh *et al.* [47].

Infrared spectroscopy further confirms that the fibers recovered after THF dissolution are composed of pure PET, devoid of any residual PVC or traces

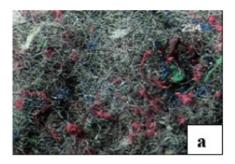




Figure 7. PET fibers recovered at "outlet F". (a) Magnification 6.3×. (b) Magnification 16×.





Figure 8. PVC powder recovered at "outlet P". (a) Magnification 6.3×. (b) Magnification 16×.



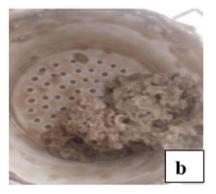


Figure 9. (a) Homogeneous mixture containing a PVC and THF solvent. (b) Recovery of insoluble PET fibers.

of solvent. This finding complements the results obtained by optical microscopy, providing a comprehensive understanding of the composition and purity of the recovered fibers.

Determination of the different amounts of PET fibers and PVC powder using chemical separation. In this case study, a 50-g sample of ground PVC-coated fabric scraps was dissolved in THF, following the previously described method. The PVC-coated

fabric scraps used in this study exhibit the characteristics listed in Table 1.

Table 5 provides a comprehensive summary of the PET fibers and plasticized PVC proportions present in the PVC-coated fabric scraps. It should be noted that all experiments were conducted three times to ensure accuracy and reliability.

According to Table 5, the analysis reveals that the PVC-coated fabric scraps contain approximately 15.77% PET fibers and 83.75% plasticized PVC.

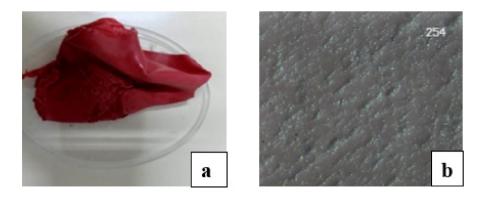


Figure 10. Plasticized PVC. (a) Magnification 6.3×. (b) Magnification 16×.



Figure 11. Insoluble knitted textile substrate of PET fibers. (a) Magnification 6.3×. (b) Magnification 16×.

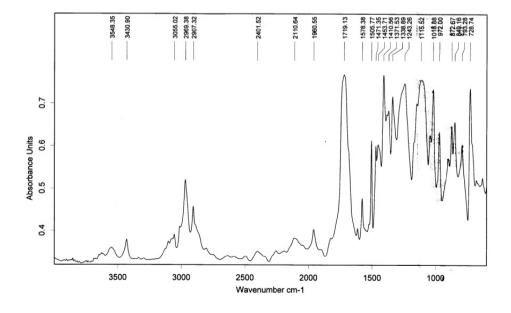


Figure 12. Infrared spectrum of poly(ethylene terephthalate).

Table 4.	Band	assignments	for the	infrared	spectrum of PET

Description of vibration (Assignment)	Wavenumbers (cm ⁻¹)
Absorbed moisture	3535
O–H stretching of diethylene glycol end-group	3440
Aromatic C–H stretching	3060
Aliphatic C–H stretching	2960, 2880
Aromatic summation band	1950
Carbonyl C=O stretching	1720
Aromatic skeletal stretching bands	1615, 1450, 1430,
- CH ₂ - deformation band	1465
C(O)–O stretching of ester group	1270
Bands in the skeletal ring region indicate 1,4-substitution	1175, 1120, and 1020
O-CH ₂ stretching of ethylene glycol segment in PET	980
C-H deformation of two adjacent coupled hydrogens on an aromatic ring	850
Associated with the out-of-plane deformation of the two carbonyl substituents on the aromatic ring $$	730

Table 5. PET fibers and plasticized PVC proportions present in the PVC-coated fabric scraps

	Average (g)	Percentage (%)	CV (%)
PET fibers	7.89	15.77	3.34
Plasticized PVC	41.88	83.75	1.35

These findings closely align with the characteristics presented in Table 1, which indicated 16% PET fibers and 84% plasticized PVC. The remarkable similarity between these two datasets serves as compelling evidence that affirms the reliability of the chemical method employed in this study.

Furthermore, Table 5 highlights a notable reduction in the total mass recovered through the implementation of the chemical method compared with the mass of the initial sample. To gain a comprehensive understanding of the underlying mechanisms causing this decrease in initial mass, it is essential to consider the thorough characteristic study already undertaken, which involved meticulous microscopic observation and Fourier Transform Infrared Spectroscopy (FTIR).

The experimental results following chemical separation demonstrate the successful recovery of the PET knitted fabric. However, it is crucial to

acknowledge that despite confirming the retrieval of highly pure plasticized PVC without any PET fibers or residual THF solvent through distillation, several studies [10,42] suggest a potential loss of a fraction of the plasticizer during the THF solvent extraction process via distillation.

This loss can be attributed to the relatively volatile nature of the plasticizer compound, which makes it prone to evaporation. As a result, when the THF solvent is distilled off, the plasticizer also evaporates, leading to a decrease in the overall mass after the chemical method.

For the purpose of our study, we can primarily focus on the initial phase of PVC dissolution in the THF solvent because the successful recovery of the PET textile substrate and determination of its percentage allow us to infer the corresponding percentage of plasticized PVC.

To determine the average weights of PET fibers and plasticized PVC in each outlet (PVC powder outlet (P) and fiber outlet (F)) after pulverization, the chemical separation method was employed. To ensure accuracy, the experiments were conducted three times.

According to the chemical separation results (Table 6), the quantity recovered from outlet P primarily comprises PVC powder, accounting for approximately 92.39%. However, a small amount of

		Average (g)	Percentage (%)	CV (%)
PVC powder outlet $(P) = 50 g$	Fibers	3.07	6.15	18.51
PVC powder outlet (P) = 50 g	PVC	46.19	92.39	2.62
Fiber outlet $(F) = 50 g$	Fibers	36.09	72.17	4.32
riber outlet (r) = 30 g	PVC	12.54	25.08	5.80

Table 6. Different amounts of PET fibers and PVC powder after pulverization

PET fibers, not exceeding 6.15%, was also detected. On the other hand, outlet F contains a higher percentage of residual PVC, reaching 25.08%, whereas the quantity of PET fibers amounts to approximately 72.17%.

These findings confirm previous observations and indicate that the mechanical grinding process fails to achieve complete separation between the powders and PET fibers. As mentioned earlier, the challenge of separation remains largely unresolved, especially when it comes to achieving complete isolation of textile fibers from plasticized PVC.

Consequently, our primary focus is to enhance the separation process between PET fibers and PVC powder to identify the most efficient method.

3.3. Sieving separation

The mechanical grinding process described earlier allows the transformation of PVC-coated fabric scraps into powders. However, it is limited by the challenge of obtaining a clean PVC powder. To optimize the separation efficiency between the PVC powder and fibers and to maintain the PVC in powder form, an additional sieving step was incorporated in conjunction with the mechanical grinding process.

The sieving process encompassed preparatory steps, including sampling and selection of an appropriate sieve [49–54]. The samples used for mechanical sieving were obtained in accordance with the previously outlined sampling procedure.

3.3.1. Selection of an appropriate sieve aperture

To achieve optimal separation, selecting an appropriate sieve with the correct aperture is crucial to ensure complete detachment of fibers from the powder. Accurate prediction of this aperture using theoretical methods or approaches is challenging because of a limited understanding of the fundamental particle motion and screen penetration rate [52,53].

Therefore, a series of sieves with various apertures were tested to assess their separation efficiencies in terms of PET fibers and PVC powder.

The sieving process (Figure 4) involved placing a 500 g sample of powder onto sieves with fixed aperture sizes (2 mm, 1 mm, 0.8 mm, 0.6 mm, 0.4 mm) and agitating the sieves with an amplitude of 1.2 mm. A receiving pan was positioned underneath all the sieves to collect the samples.

Subsequently, the efficacy of this sieving method in separating PET fibers from PVC powder was tested for different durations (2, 5, and 10 min). Initial observations for each sieving time (2, 5, and 10 min) revealed that the top sieve with the largest aperture size (2 mm) retained only the fiber clumps, allowing all other particles to pass through the sieve apertures (Figure 13). However, the subsequent sieve with an aperture size of 1 mm retained only a small amount of PVC powder and fiber clumps (Figure 14).

The experimental observations can be attributed to the phenomenon of entangled fibers forming agglomerates through interlocking, which facilitates their separation via vibratory sieving.

During vibratory sieving, particles experience a three-dimensional movement that combines circular motion with vertical throwing motion. These combined motions result in increased interactions and cohesive bonds between PET fibers, primarily because of their unique shapes [55]. According to Blancoa *et al.* [56], this cohesion leads to the formation of Van der Waals forces (dipole–dipole interaction) and frictional effects caused by electrostatic charges or forces.

As mentioned earlier, due to the cohesive forces and interlocking nature of the fiber clumps, several of them cannot pass through the uppermost sieve (2 mm) because their sizes exceed the mesh size of this particular sieve.

Regarding the remaining sieves (Figure 15) with aperture sizes of 0.8, 0.6 and 0.4 mm, as well as the





Figure 13. Fiber clumps collected on a sieve (aperture = 2 mm).



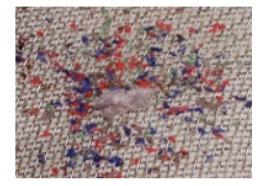


Figure 14. Fiber clumps with powder collected on a sieve (aperture = 1 mm).

receiving pan, microscopic examinations revealed the presence of PVC powders alone, without any fibers. These findings were further substantiated using the chemical separation method. Specifically, after subjecting the samples to treatment with tetrahydrofuran (THF) solvent, no trace of fibers was recovered, and all of the powder demonstrated solubility in the solvent.

Therefore, a single sieve with a 1-mm aperture size can effectively separate the "oversized" fiber amount from the "undersized" PVC powder.

3.3.2. Verification of the complete separation procedure

To achieve comprehensive separation and ensure reliable reproducibility, an additional step was incorporated alongside the mechanical grinding process. A simple sieving in motion, utilizing a sieve with a 1-mm aperture, was conducted for 5 min. The sieve was placed on top of a collecting pan to gather the separated materials (Figure 16).

To evaluate the efficiency of the separation process, a representative sample weighing 50 g from the powder outlet (P) was sieved using a standard sieve with a 1-mm aperture. Subsequently, the oversize and undersize portions obtained from the sieving process were subjected to chemical testing. The corresponding results are presented in Table 7. It is worth noting that all experiments were conducted in triplicate to ensure reliability and consistency.

The experimental findings presented in Table 7 demonstrate that, following the sieving process, approximately 45.92 g (equivalent to around 91.84%) of PVC powder was successfully recovered in the collecting pan (undersize), while all fibers were retained (3.42 g, approximately 6.84%) by the 1 mm mesh sieve, indicating that a complete separation between PET fibers and PVC powder was achieved.

These results align with those obtained from the chemical separation analysis (Table 6), confirming that the quantity of residual fibers in the recovered PVC powder at the outlet (P) of the pulverizer was approximately 6%.

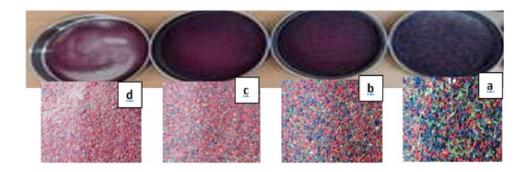


Figure 15. Retained powder with different aperture sizes: (a) 0.8 mm, (b) 0.6 mm, (c) 0.4 mm, (d) pan.

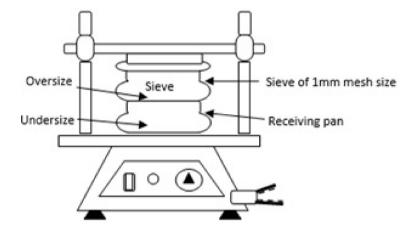


Figure 16. Simple sieving (aperture = 1 mm).

Table 7. Different amounts of PET fibers and PVC powder after sieving

		Average (g)	Percentage (%)	CV (%)
Oversize	Total oversize weight	3.68	100	8.94
	PVC	0.25	6.98	18.41
	Fibers	3.42	93.02	8.49
Undersize	Total undersize weight	45.92	100	0.49
	PVC	45.92	100	0.49
	Fibers	0	0	

The combination of the mechanical process (cutting, grinding, pulverization) and sieving (Figure 17) proves to be a straightforward, cost-effective, environmentally friendly, and potentially profitable approach. Moreover, this method effectively addresses both objectives: (1) separating fibers from PVC, and (2) maximizing the recovery of small PVC powders.

The employed methods, namely microscopic observation and chemical separation, serve the initial purpose of validating the efficacy of the preceding process in successfully obtaining finely separated PVC particles from the fibers. The subsequent crucial aspect involves evaluating the yield of this process, specifically determining the percentage of recovered

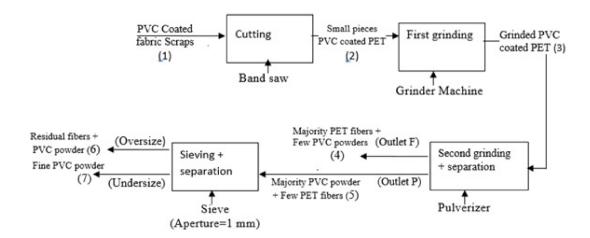


Figure 17. Process map of grinding supplemented by sieving.

PVC powder in relation to the initial quantity of PVC introduced into the process.

3.4. Process yield

Process yield, which is a widely accepted practice, provides a valuable metric for assessing process performance, typically quantified using Equation (1).

Process Yield =
$$\frac{Q(PVC)out}{Q(PVC)in}$$
 (1)

Q(PVC) out: Quantity of PVC recovered at the end of the process.

Q(PVC) in: Initial quantity of PVC entering the process.

This metric ensures that the recovered PVC quantity is neither entangled with the fibers nor "lost" within the machinery (pulverizer, sieve).

For this particular case study, the PVC-coated PET scrap (10 kg) possesses the characteristics outlined in Table 1. To demonstrate the overall effectiveness of the process, Table 8 presents the total mass of recovered PVC powder after pulverization and subsequent sieving. To ensure statistical validity, the process is repeated three times, yielding reliable and conclusive results.

To adhere to the maximum sieved material limit of 3 kg for these test sieves, three passes are required for each test to ensure the complete sieving of all PVC powder derived from pulverizer outlet P. To provide a clearer visualization of the results, the following process map has been included.

The results demonstrate that approximately 7.05 kg of PVC powder is successfully recovered at the end of the process, following the sieving step. This amount corresponds to a percentage of 83.92% in comparison with the initial PVC quantity (approximately 8.4 kg). Such high efficiency (0.839) is particularly promising and encourages further development of this process on an industrial scale.

Regarding the overall process, there is a difference of approximately 1.35 kg (8.4-7.05), representing approximately 16.07%. This discrepancy results from both agglomeration with the fibers and significant retention within the pulverizer.

In addition, it should be noted that the quantity of PET fibers recovered at outlet 6 (as depicted in Figure 18) is combined with the amount retrieved at outlet 4.

The aforementioned experimental findings will be further explored in subsequent publications to leverage the potential of pulverized PVC-coated fabric scraps. Specifically:

- Recovered PVC powder (outlet (7), Figure 18):
 The successful separation of PVC powder enables its reuse as a substitute for PVC resin in plastisol, particularly for use in floor coverings. Detailed results pertaining to the process of PVC-coated fabrics derived from PVC powder waste will be presented in a forthcoming study.
- Recovered fibers (outlets (4 + 6), Figure 18): These PET fibers, along with residual traces

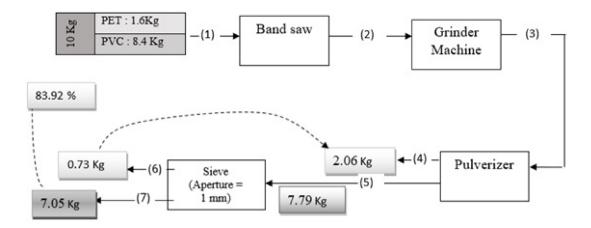


Figure 18. Schematic of the recovery process.

Table 8. Different amounts of PET fibers and PVC powder during complete mechanical separation

			Average (kg)	Percentage (%)	CV (%)
	Input weight		10	100	
Pulverizer	Output weight	Outlet F (4)	2.06	20.63	12.8
		Outlet P (5)			
	Input weight		7.79	77.87	3.41
Sieve (aperture = 1 mm)	Output weight	Oversize (6)	0.73	9.34	4.91
		Undersize (7)	7.05	90.58	3.57

of PVC, can be repurposed as a binder under high-temperature conditions to produce nonwoven materials through a melt process. These initial findings are currently undergoing laboratory testing and will be further elucidated in subsequent studies that explore diverse applications for these materials.

4. Conclusion

This investigation focuses on the mechanical recycling process of PVC-coated fabric scraps. The technique employed involves a grinding process consisting of three main steps: cutting, first grinding, and second grinding. These steps enable the transformation of PVC-coated fabric scraps into PVC powder while partially separating them from the PET fibers.

The effectiveness and yield of this process are discussed through qualitative and quantitative analyses.

The qualitative analysis, conducted using microscopic observation, reveals that the PET fibers from

"outlet F" are entangled with the PVC particles, posing challenges for their mechanical separation. In contrast, the quantitative analysis utilizes tetrahydrofuran (THF) to dissolve the plasticized PVC, providing precise values such as the exact percentage of each constituent following the grinding process.

Combining the qualitative and quantitative analysis methods, it can be concluded that the separation between PET fibers and the PVC powder recovered from both pulverized outlets (outlet P and outlet F) is not perfect, even though the percentage of PET fibers in the powder does not exceed 6.15% for outlet P. The presence of PVC alongside PET fibers significantly compromises the quality of the recycled material because of the distinct properties of these two components. Consequently, the grinding process alone is insufficient for the reuse of PVC-coated fabric scraps.

To achieve complete separation between the PVC powder and PET fibers, supplementary mechanical sieving using a 1-mm aperture sieve was

implemented. This efficient separation was corroborated by chemical analysis, which demonstrated that approximately 90.58% of the PVC powders were successfully recovered through sieving, whereas all the PET fibers were retained by the sieve. These results provide strong evidence of the achievement of complete separation of the PVC powder.

Finally, the study reveals that while there is an overall loss of PVC in the process, approximately 16.07% is either lost through agglomeration with the fibers or retained within the pulverizer. However, it is noteworthy that 83.92% of the initial PVC was effectively recovered as powder by the end of the process, providing a valuable starting point for the development of new products. This substantial recovery of PVC powder confirms the economic feasibility and viability of the proposed method.

Future research should also consider understanding the morphology and size distribution of powder particles, as these factors are crucial prerequisites for various manufacturing operations. Morphology and size distribution have a significant impact on the functional properties of the powder, such as density and flowability. Therefore, studying these characteristics is essential for optimizing the manufacturing processes and ensuring desirable functional properties of the powder.

Declaration of interests

The authors do not work for, advise, own shares in, or receive funds from any organization that could benefit from this article, and have declared no affiliations other than their research organizations.

References

- [1] J.-J. Sion, J. Coat. Fabr., 1998, 28, 73-79.
- [2] W. Zhang, X. Ding, X. Yang, Adv. Mater. Res., 2011, 160, 1476-1481.
- [3] S. Gargoubi, W. Chaouch, M. Stambouli, N. Bhouri, C. Boudakhane, R. Zouari, Chem. Ind. Chem. Eng. Q., 2019. 25, 141-151.
- [4] J. H. Eun, M. S. Kim, S. M. Sung, B. K. Choi, I. U. Jang, D. Kim, J. S. Lee, J. Ind. Text., 2020, 51, 2897-2920.
- [5] K. Singha, Am. J. Polym. Sci., 2012, 2, 39-49.
- [6] G. Blenkhorn, J. Coat. Fabr., 1998, 28, 93-105.
- [7] Y. Yu, Z. Cao, Y. Sun, Structures, 2021, 33, 830-840.
- [8] M. Stambouli, W. Chaouch, S. Gargoubi, R. Zouari, S. Msahli, Turkish J. Chem., 2023, 47, 40-46.
- [9] M. Stambouli, W. Chaouch, S. Gargoubi, R. Zouari, E. Visileanu, S. Msahli, *Ind. Textile*, 2022, 73, 580-586.

- [10] R. Saffert, J. Coat. Fabr., 1994, 23, 274-279.
- [11] M. Sadat-Shojai, G.-R. Bakhshandeh, *Polym. Degrad. Stab.*, 2011, 96, 404-415.
- [12] S. Mark Gillette, J. Coat. Fabr., 1992, 22, 75-79.
- [13] G. Wu, J. Li, Z. Xu, Waste Manag., 2013, 33, 585-597.
- [14] D. Braun, Prog. Polym. Sci., 2002, 27, 2171-2195.
- [15] T. Kameda, Y. Fukuda, G. Grause, T. Yoshioka, J. Appl. Polym. Sci., 2010, 116, 36-44.
- [16] M. A. Keane, ChemSusChem, 2009, 2, 207-214.
- [17] J. N. Hahladakis, C. A. Velis, R. Weber, E. Iacovidou, P. Purnell, J. Hazard. Mater., 2018, 344, 179-199.
- [18] T. Kameda, Y. Fukuda, G. Grause, T. Yoshioka, J. Appl. Polym. Sci., 2010, 116, 36-44.
- [19] D. Garcia, R. Balart, J. E. Crespo, G. Juan Lopez, J. Appl. Polym. Sci., 2006, 101, 2464-2471.
- [20] D. Pan, F. Su, C. Liu, Z. Guo, Adv. Compos. Hybrid Mater., 2020, 3, 443-461.
- [21] F. Burat, A. Güney, M. Olgaç Kangal, Waste Manag., 2009, 29, 1807-1813
- [22] M. A. Keane, J. Chem. Technol. Biotechnol., 2007, 82, 787-795.
- [23] G. Wu, J. Li, Z. Xu, Waste Manag., 2013, 33, 585-597.
- [24] E. A. Williams, P. T. Williams, J. Chem. Technol. Biotechnol., 1997, 70, 9-20.
- [25] M. Abbasi, M. M. Salarirad, I. Ghasemi, *Iran Polym. J.*, 2010, 19, 483-489.
- [26] K. Ragaert, L. Delva, K. Van Geem, Waste Manag., 2017, 69, 24-58.
- [27] J. Pan, H. Jiang, T. Qing, J. Zhang, K. Tian, Chemosphere, 2021, 284, 1-10.
- [28] J. Zhou, G. Liu, S. Wang, H. Zhang, F. Xu, J. Energy Inst., 2020, 93, 2362-2370.
- [29] Q. Ma, Z. Wu, V. A. Neacşu, S. Zhao, Y. Chai, H. Zhang, *Nano Res.*, 2022, 15, 9921-9925.
- [30] W. S. Fonseca, X. Meng, D. Deng, ACS Sustainable Chem. Eng., 2015, 3, 2153-2159.
- [31] G. Grause, A. Buekens, Y. S. A. Okuwaki, T. Yoshioka, J. Mater. Cycles Waste Manag., 2011, 13, 265-282.
- [32] D. Damayanti, D. R. Saputri, D. S. S. Marpaung, F. Yusupandi, A. Sanjaya, Y. M. Simbolon, W. Asmarani, M. Ulfa, H.-S. Wu, Polymers, 2022, 14, 1-29.
- [33] M. G. Davidson, R. A. Furlong, M. C. McManus, J. Clean. Prod., 2021, 293, article no. 126163.
- [34] M. M. Aji, S. Narendren, M. K. Purkait, V. Katiyar, J. Environ. Chem. Eng., 2020, 8, article no. 103650.
- [35] I. A. Ignatyev, W. Thielemans, B. Vander Beke, *ChemSusChem*, 2014, 7, 1579-1593.
- [36] C. Gutiérrez, M. T. García, I. Gracia, A. de Lucas, J. F. Rodríguez, Waste Biomass Valor., 2013, 4, 29-36.
- [37] M. N. Siddiqui, J. Hazard. Mater., 2009, 167, 728-735.
- [38] S. M. Al-Salem, P. Lettieri, J. Baeyens, Prog. Energy Combust. Sci., 2010, 36, 103-129.
- [39] J. M. Garcia, M. L. Robertson, Science, 2017, 358, 870-872.
- [40] M. Paci, F. P. La Mantia, Polym. Degrad. Stab., 1999, 63, 11-14.
- [41] M. S. Negari, S. O. Movahed, A. Ahmadpour, Sep. Purif. Technol., 2018, 194, 368-376.
- [42] S. Adanur, Z. Hou, R. M. Broughton, J. Coat. Fabr., 1998, 28, 145-168.
- [43] S. Adanur, Z. Hou, R. M. Broughton, J. Coat. Fabr., 1998, 28, 37-55.

- [44] L. C. Lerici, M. S. Renzini, L. B. Pierella, Proc. Mater. Sci., 2015, 8, 297-303.
- [45] G. Grause, S. Hirahashi, H. Toyoda, T. Kameda, T. Yoshioka, J. Mater. Cycles Waste Manag., 2017, 19, 612-622.
- [46] B. J. Holland, J. N. Hay, Polymer, 2002, 43, 1835-1847.
- [47] S. Ramesh, K. H. Leen, K. Kumutha, A. K. Arof, Spectrochim. Acta A: Mol. Biomol. Spectrosc., 2007, 66, 1237-1242.
- [48] C. Wang, H. Wang, Y. Liu, J. Clean. Prod., 2015, 102, 378-383.
- [49] U. Knauf, A. Maürer, W. Holley, M. Wiese, H. Utschick, *Kunstst. Plast Eur.*, 2000, 90, 24-26.
- [50] C. S. Liu, J. X. Lu, J. China Coal Soc., 2009, 34, 556-559.

- [51] Y. M. Zhao, C. S. Liu, J. China Coal Soc., 2000, 25, 206-208.
- [52] J. Li, C. Webb, S. S. Pandiella, G. M. Campbell, *Powder Technol.*, 2003, 133, 190-202.
- [53] P. W. Cleary, M. D. Sinnott, R. D. Morrison, *Miner. Eng.*, 2009, 22, 1218-1229.
- [54] B. Chen, J. Yan, W. Mo, C. Xu, L. Zhang, K. K. Tamma, J. Vibroengineering, 2019, 21, 2025-2038.
- [55] D. Hailin, L. Chusheng, Z. Yuemin, Z. Lala, *Int. J. Mining Sci. Technol.*, 2013, 23, 95-98.
- [56] D. Blancoa, O. Antikainen, H. Räikkönen, P. T. Mah, A. M. Healy, A. M. Juppo, J. Yliruusi, *Int. J. Pharm.*, 2020, 581, article no. 119280.