

Article

Recovery and Reuse of Acetone from Pharmaceutical Industry Waste by Solar Distillation

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Abstract: Solvents are particularly hazardous among the mixture of pollutants found in the air, as their low vapor pressure allows them to reach the atmosphere, causing damage to ecosystems, and producing secondary deleterious effects on living organisms through a wide variety of possible reactions. In response, innovative, sustainable, and ecological methods are being developed to recover solvents from industrial wastewater, which is typically contaminated with other organic compounds. This study describes the procedure for recovering acetone from a residue from the pharmaceutical industry. This compound contains a high amount of solid organic compounds, which are generated during the manufacture of medicines. The treatment consisted of performing a simple solar distillation using a single-slope glass solar still, which separated the acetone from the mother solution. Under ideal circumstances, the use of solar radiation allowed an efficiency rate of 80% using solar concentration by means of mirrors to increase the temperature and 85% without the use of mirrors in the production of distilled acetone, which was characterized to evaluate its quality using instrumental analytical techniques: NMR, IR, and GC. The results obtained indicate that the acetone recovered by this procedure has a good quality of 84%; however, due to this percentage obtained, its reuse is limited for certain applications where a high degree of purity is required, such as its reuse for pharmaceutical use; for this reason, it was proposed to use said compound to eliminate the organic impurities contained in the catalyst waste granules used in a Mexican oil refinery. The resulting material was examined by SEM and EDS, revealing a high initial carbon content that decreased by 29% after treatment. Likewise, as an additional study, a study was carried out to evaluate the characteristics of the residues obtained at the end of the distillation where rubidium, silicon, carbon, nitrogen, oxygen, and chlorine contents were observed.

Keywords: acetone; solar distillation; reuse



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1. Introduction

Waste generated by the pharmaceutical industry poses a major problem when it comes to being dumped into the environment, due to the wide variety of compounds it contains, and has been classified as potentially hazardous waste. Its presence in the environment damages natural resources and living beings, and interferes with nature's

biochemical processes [1,2]. Substances classified in this category include organic solvents such as acetone, which is used in a wide variety of processes, from which a large amount of potentially harmful waste is generated [3,4]. Acetone has several uses, such as a raw material, viscosity modifier, surfactant, plasticizer, and impurity remover. However, this compound is usually dispersed in the biosphere and contributes 35% of the total volatile organic compounds (VOCs) [5]. The production of petrochemicals such as acetone is highly sought after due to its diverse applications and indispensable role in the manufacturing of products, including the manufacture of paints, varnishes, lacquers, pharmaceuticals, and cosmetics [6,7]. Acetone is produced by the process of cumene peroxidation or by the dehydrogenation of isopropyl alcohol (2-Propanol). However, these methods carry economic and environmental disadvantages due to the use of high temperatures and pressures [8,9]. In addition to this, according to a recent report, industrialized countries adequately treat less than 4% of solvents, although 87.4% are recyclable [10], leading to their incineration and an increased carbon footprint for the chemical industry. Therefore, it is essential to develop sustainable and environmentally friendly techniques that are efficient in the recovery of organic solvents (since these usually escape from the systems due to their low boiling point) that are often mixed with other substances, to prevent them from being released into the atmosphere and to ensure compliance with quality standards for their reuse [11].

Solvent recovery in pharmaceutical companies has been recently studied with the aim of increasing the percentage of recovery and reuse, resulting in a minimized reduction in greenhouse gas emissions [3]. A recent study indicates that through liquid–liquid extraction, methyl isobutyl ketone and ethyl methyl acetone can be recovered in a high percentage. The main drawback is that sodium hydroxide extraction is required to achieve the desired purity, so waste is generated in the process [12]. For this reason, the use of renewable energies provides important energy, economic, and environmental benefits [12–14]. In this sense, solar distillation has proven to be a favorable technique in the recovery of compounds, due to its high efficiency [15].

Table 1 presents a comparison of the acetone recovery rates obtained using various methods reported in the literature and the one proposed in the present study. The results show that solar distillation emerges as an efficient and sustainable alternative, characterized by its high efficiency and low energy requirements. It is important to note that the information available on the application of solar distillation for solvent recovery is limited, which further underscores the relevance and innovative nature of this work.

Table 1. Comparison of Acetone Recovery Rates.

Method	Recovery Rate	Reference
Separation of the components of the azeotropic mixture acetone– <i>n</i> -hexane by a combined process: liquid–liquid extraction and batch distillation	87.9%	[16]
Evaluation and analysis of the separation and purification system for the acetone–butanol–ethanol mixture using liquid–liquid distillation/extraction sequences	98.88%	[17]
Separation of the azeotropic mixture acetone–methanol with lithium chloride as an extractive agent	88.85%	[18]
This project:		
Solar distillation	85%	
Solar distillation–concentration	80%	-

Solar stills offer some advantages, as they can be constructed using various materials, which determine their durability and operating capacity. They are also easy to manufacture, require minimal labor for installation, and need little maintenance [19,20]. The efficiency of

these devices also depends on factors such as the geometry, location, orientation, incidence of electromagnetic radiation, and ambient temperature [21–24].

In 2020, Chávez and collaborators analyzed both types of solar distillation processes using internal reflectors with a solution of deionized water with 70% ethyl alcohol, with a total volume of 1 L. They also tested mixtures with 200/800, 400/600, 600/400, 800/200, and 1000/0 mL of alcohol. Solar distillation proved to be a viable method for alcohol recovery, achieving an increase in concentration from 11 to 14 degrees of alcohol within the first hour and distilling, on average, 80% of the mixture, regardless of the alcohol content [25].

Given the advantages of the proposed method, in this project, a solar concentration–distillation system was used for treating a residue from the pharmaceutical industry. This residue contains a mixture of pure acetone with organic solid compounds used during the crystallization of substances used as active ingredients. Among these, clonazepam was found in the highest concentration. Table 2 presents the volatility of the compounds identified in the characterization of the pharmaceutical residual liquid. The objective was to evaluate the efficiency of a sustainable device designed for acetone recovery and to determine its quality parameters. An analysis of the acetone distilled via solar distillation revealed a purity level of 84%. However, for reuse in the pharmaceutical industry, acetone typically requires a purity level of over 99.5%, as specified by regional pharmacopoeias, such as the USP (United States Pharmacopoeia), EP (European Pharmacopoeia), and WHO (International Pharmacopoeia), which set strict limits on allowable impurities to ensure compliance with the quality and safety standards for use in formulations or the cleaning of medical and laboratory equipment. For this reason, it was proposed to reuse the recovered acetone in a different sector, specifically, for removing impurities from the granules of a catalyst used in a refinery to convert heavy oil into lighter products [26,27]. Additionally, an evaluation of the solids obtained from the separation of acetone at the end of the distillation was conducted.

Table 2. Volatility of Components Found in Pharmaceutical Residual Liquid.

Compound	Vapor Pressure at 20 °C	Volatility Level	Reference
Acetone	233 mmHg	High	[28]
Chlorine	5160 mmHg	High	
Carbon	0 mmHg	Does not volatilize	[29]
Nitrogen	0.12 mmHg	Does not volatilize	
Oxygen	0.38 mmHg	Does not volatilize	

2. Materials and Methods

2.1. Solar Distillation

A single-slope solar still made of smooth, distortion-free glass was designed and constructed, featuring a black stainless steel tray as the solar heat collector, with a maximum capacity of 500 mL. The design incorporated a 20° slope to optimize solar radiation capture. The device included an inlet for pharmaceutical residual liquid and an outlet for the distilled solvent, which was extracted using a 5 W and 12 V peristaltic pump, brand K Kamoer NKP, which is powered by a 30 W and 12 V solar panel, brand Solar Charger, and a flexible silicone hose, brand Walfront, resistant to UV rays and high and low temperatures, with an internal diameter of 2 mm, and collected in amber flasks (Figure 1a). Subsequently, mirrors with 3× magnification were installed as solar concentrators to enhance solvent (acetone) recovery (Figure 1b).

In order to determine the efficiency of the device, two experiments were conducted: one with solar concentration and one without. Both experiments were carried out in Toluca, State of Mexico, Mexico, located at a latitude of 19°17'29".

Operating principle: The distiller was oriented towards the equator to maximize solar radiation capture, which was absorbed by the tray. In the case of Figure 1b, the mirrors enhanced the temperature through solar concentration, directing heat towards the container to raise the temperature of the pharmaceutical residual liquid inside. Following the evaporation–condensation, the recovered solvent was pumped to the outlet and collected in containers (the blue arrows indicate the route followed by the distilled acetone recovered in the still until it reaches the collector).

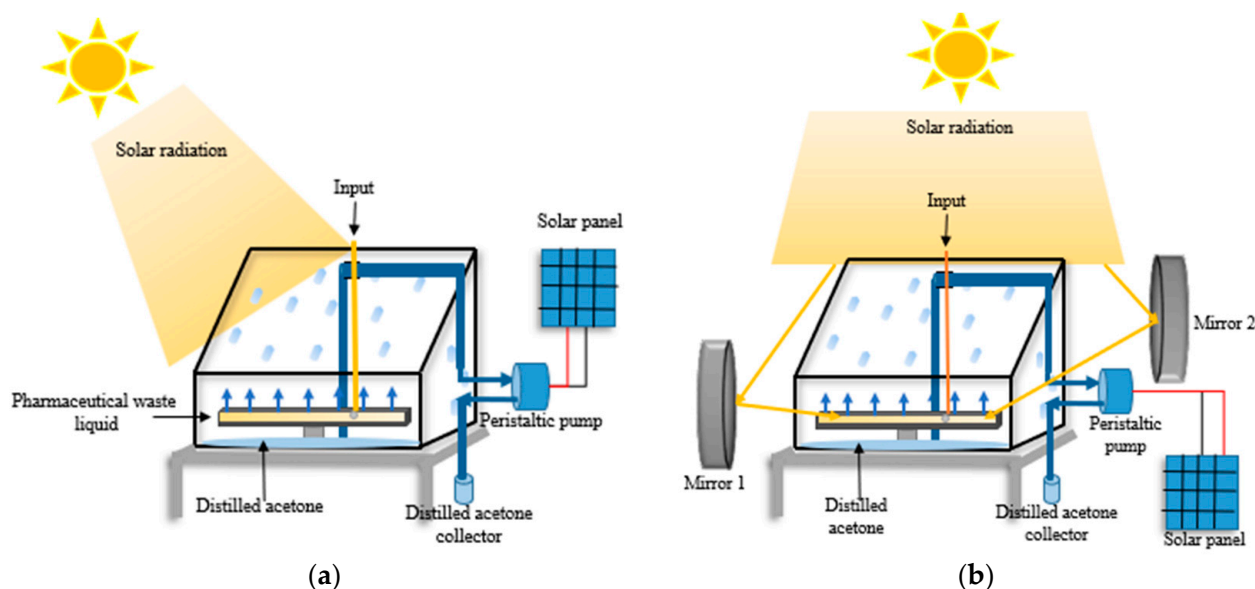


Figure 1. Solar still: (a) without mirror adaptation, and (b) with mirrors as solar concentrators.

Solar Distiller Efficiency Evaluation

Two experiments were conducted between May and July 2024 to evaluate the temperature and the amount of acetone recovered at the end of the process. The goal was to establish the optimal conditions for obtaining a higher volume of distilled acetone.

In the first experiment, a 250 mL sample was placed inside the solar still at 11:00 a.m., with an initial temperature of 20.0 °C. Temperature measurements were recorded every 10 min until the liquid had completely evaporated. At that point, the volume of the collected acetone was measured using a test tube to determine the efficiency of the distiller.

In the second experiment, the same amount of acetone was placed in the distiller under the operating conditions described above. However, mirrors were added to act as solar concentrators, increasing the temperature of the residual liquid to be distilled. As in the previous case, temperature measurements were taken every 10 min until the liquid had completely evaporated, and the volume of acetone collected was measured to determine the efficiency of the distiller adapted with solar concentration.

The efficiency of each experiment was calculated using Equation (1).

$$Efficiency = \frac{Vol.distilled\ acetone}{Vol.Initial\ residual\ liquid} \times 100 \quad (1)$$

2.2. Characterization of Distilled Acetone

The characteristics of the distilled acetone at the end of the process were evaluated using the following analytical techniques:

2.2.1. Nuclear Magnetic Resonance

The molecular structure and chemical composition of the distilled sample were determined using nuclear magnetic resonance (NMR) spectroscopy, with a Bruker Avance model 300 MHz spectrometer equipped with 7.04 T shielded magnets. Calibration was performed using residual CHCl_3 in the solvent used (7.27 ppm for ^1H NMR and 77 ppm for ^{13}C NMR). A total of 16 pulses were programmed for ^1H NMR signal accumulation and 128 for ^{13}C NMR.

2.2.2. FTIR Spectroscopy

The functional groups present in the sample were identified using infrared spectroscopy, for which a Bruker Tensor 27 Fourier Transform Infrared Spectrometer was used, with an MIR source and the ATR accessory, model Platinum ATR, Bruker, with a diamond crystal window. The spectra obtained corresponded to the infrared region (4000 to 400 cm^{-1}).

2.2.3. Gas Chromatography

An Agilent 6890 gas chromatograph equipped with a flame ionization detector (FID) and thermal conductivity detector (TCD) was used to determine the different volatile components of the distilled acetone. The analysis conditions were AT-AQUAWAX Column: $30\text{ m} \times 0.25\text{ mm} \times 0.25\text{ }\mu\text{m}$, Temp: $150\text{ }^\circ\text{C}$ and FID Temp: $200\text{ }^\circ\text{C}$, and oven ramp: starting at $40\text{ }^\circ\text{C}$ [3 min] at $10\text{ }^\circ\text{C}/\text{min}$ up to $70\text{ }^\circ\text{C}$.

2.3. Process of Removing Impurities from Catalyst Granules Using Distilled Acetone Obtained by Solar Distillation

After evaluating the distilled acetone obtained via solar distillation, it was used to remove impurities from the granules of a catalyst employed in a refinery for converting heavy oil into lighter products. In this sense, 20 mL of the distilled solvent was added to the granules (1 g) and stirred for 3 h at room temperature. The mixture was cooled to room temperature, filtered, washed with the same cold solvent, and dried under vacuum. Finally, the catalyst was treated at $650\text{ }^\circ\text{C}$ in a muffle furnace for 15 min, then cooled to room temperature for subsequent characterization [24].

The characterization of the catalyst granules, both before and after cleaning with recovered acetone, was performed using SEM/EDS to compare morphology and surface impurities. For this purpose, a NeoScope JCM-6000 scanning electron microscope was used. Images were obtained at a resolution of $3400\times$. For the EDS analysis was used a microscope from JEOL model JSM 6510LV that operates at 15 kV with an Oxford PentaFetx5 probe adapted to the SEM.

2.4. The Evaluation of the Residue Obtained at the End of the Solar Distillation Process

The composition analysis of the solid residue obtained at the end of the acetone recovery process by solar distillation was conducted using a spectrometer of X-ray photoelectron from JEOL JPS-9200 using the standard aluminum X-ray source. The spectra were acquired with 15 scans with a pass energy of 20 kV. A Shirley-type model was applied to adjust the baseline, excluding satellite peaks.

Additionally, an EDS analysis of the residue surface was performed using a NeoScope JCM-6000 scanning electron microscope to quantify the organic compounds present in the sample.

And X-ray powder diffraction was performed using a Bruker D8 advance Diffractometer using monochromated $\text{Cu K}\alpha$ radiation (X-ray source with 2.2 kW, 40 kV, and 40 mA) to identify the crystalline phases in the residual material. Diffractograms were acquired in the range of 2 theta degrees from 2 to 80° with a step of 0.02° .

3. Results and Discussion

3.1. Solar Distillation

The Evaluation of the Efficiency of the Solar Still

Figure 2 shows the data obtained from the distillation treatment with and without solar concentration. In both cases, the temperature starts at 22 °C and increases, reaching a maximum value of 68.0 °C with solar concentration and 57.3 °C without concentration, within 60 min. At this point, the distilled volume was collected and measured in a test tube, obtaining 200 mL for the first experiment and 213 mL for the second, from a 250 mL sample. The reported efficiencies were 80 and 85%, respectively. It is important to note that the solar concentration parameter resulted in a 5% decrease in efficiency, as the temperature exceeded the boiling point of acetone, causing part of it to evaporate rather than condense within the distiller, due to a reduced temperature differential.

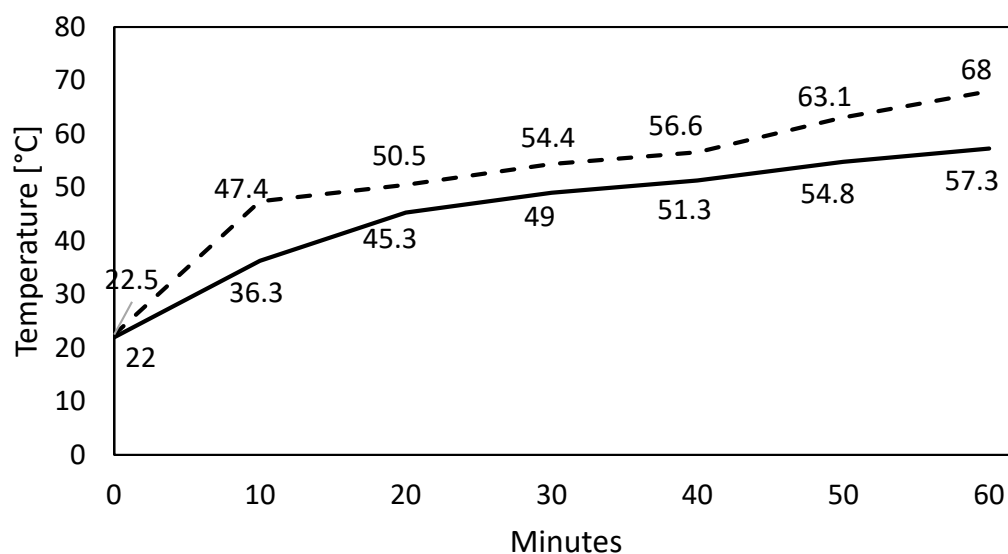


Figure 2. The determination of the temperature in the solar still without mirror adaptation (-) and with mirrors (- -) as solar concentrators.

3.2. Characterization of Distilled Acetone

3.2.1. Nuclear Magnetic Resonance

Figure 3 shows the NMR spectrum of distilled acetone, where a peak at 2.09 ppm can be observed (Figure 3a, ^1H NMR spectrum), which corresponds to the CH_3 of acetone. In the ^{13}C NMR analysis (Figure 3b, ^{13}C NMR spectrum), the aliphatic carbon of the CH_3 group is distinguished at 30.77 ppm, while the $\text{C}=\text{O}$ is located at 206.89 ppm. It should be noted that no additional signals were detected that indicate a purity higher than 84%.

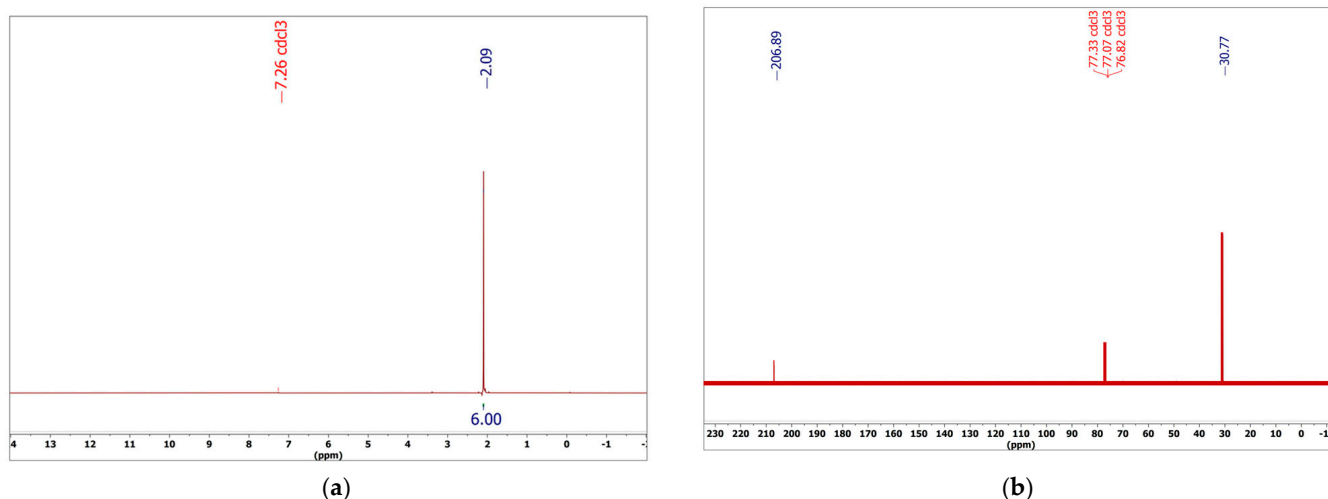


Figure 3. Nuclear magnetic resonance analysis of distilled acetone: (a) ^1H NMR, 300 MHz; (b) ^{13}C NMR, 75 MHz.

3.2.2. FTIR Spectroscopy

The structural analysis of the distilled acetone obtained by solar distillation is shown in Figure 4. Absorption bands at 2958, 2926, and 2859 cm^{-1} , corresponding to the deformation of the C-H bonds in the CH_3 groups, can be observed. Additionally, peaks at 1728 cm^{-1} indicate C=O interactions (ketone), while peaks at 1462 and 1376 cm^{-1} correspond to CH_3 vibrations. Peaks were also recorded at the wave numbers of 1269 and 1072 cm^{-1} associated with the C-H bond, as well as at 1125, 1033, and 980 cm^{-1} , attributed to the C-C bond. This is a common characteristic of simple ketones like acetone, typically appearing in this region due to the interactions between carbonyl and methyl groups. Finally, peaks at 803, 743, and 700 cm^{-1} correspond to the CH bond.

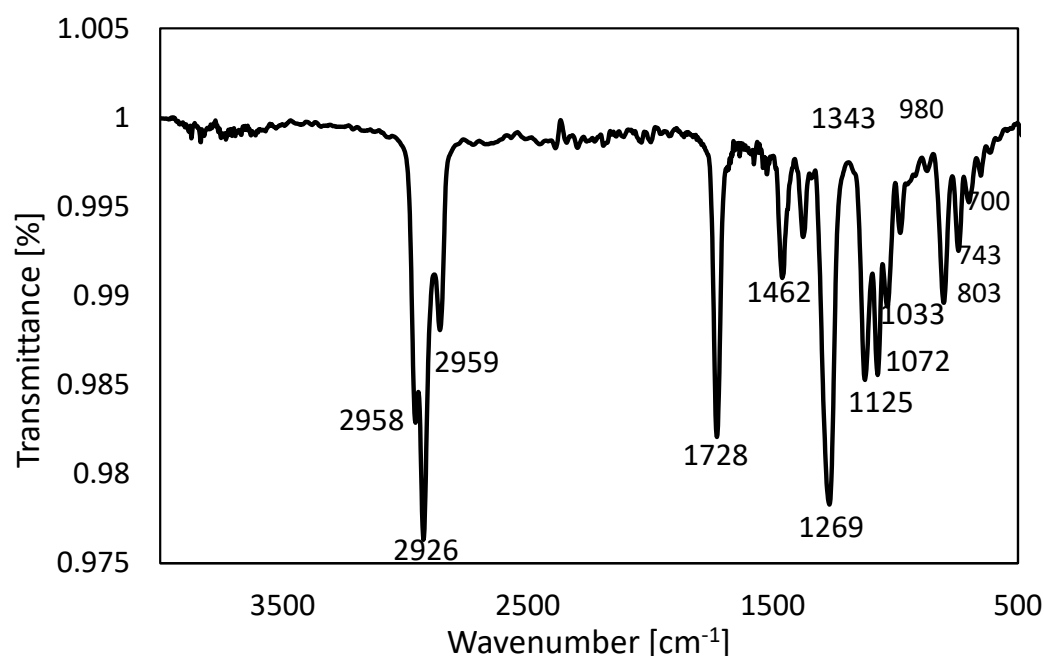


Figure 4. IR absorption spectrum obtained for distilled acetone.

3.2.3. Gas Chromatography

The gas chromatography analysis (Figure 5) of the acetone obtained through solar distillation identified three components: acetone at a retention time of 1.401 min (Peak 1),

ethyl acetate at 1.836 min (Peak 2), and water at 4.087 min (Peak 3). In addition, the area percentages of each component were as follows: 84.16% for acetone, 1.68% for ethyl acetate, and 14.15% for water.

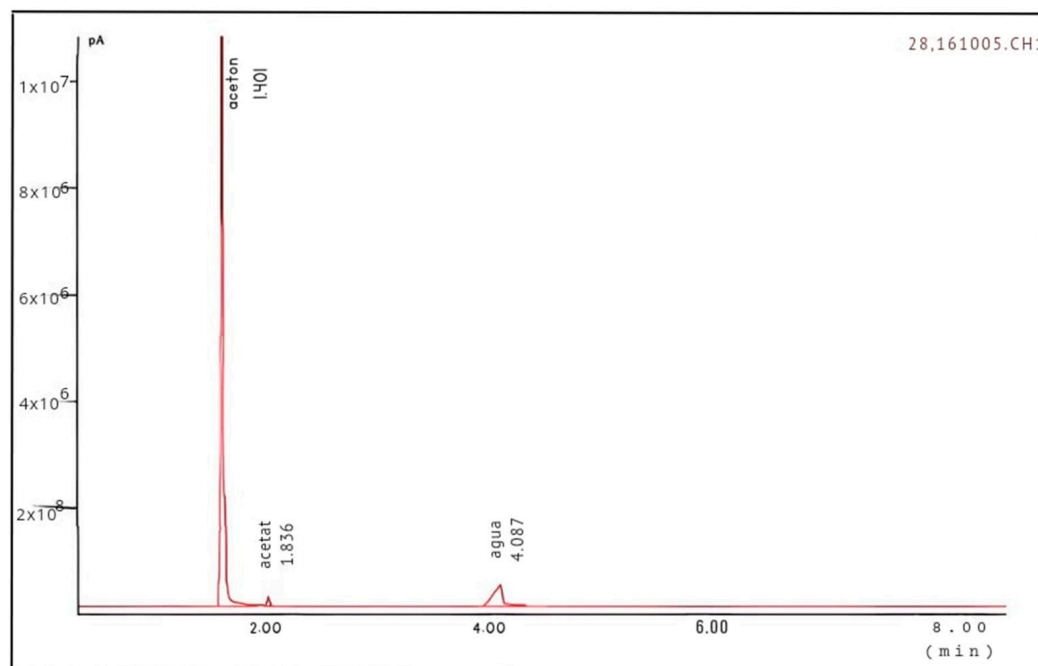


Figure 5. Gas chromatography of distilled acetone.

3.3. Process of Removing Impurities from Catalyst Granules Using Distilled Acetone from Solar Distillation

Figure 6a1,a2 present SEM micrographs showing the morphology of the catalyst surface before and after treatment for hydrocarbon removal using acetone recovered through solar distillation. The images reveal the presence of some compounds adhering to the catalyst surface. After treatment with acetone and subsequent calcination, a slight physical change is observed on the surface. The corresponding energy-dispersive spectra (Figure 6b1,b2) indicate that the untreated catalyst contains a high carbon content of 78.92% associated with hydrocarbons. Following treatment, the carbon content decreases by 29%, as evidenced in the spectra before and after treatment. The spectra also reveal the presence of S, V, and Fe peaks. These results align with previous studies, which report the deposition of V and S from heavy crude oil onto the catalyst during the reaction, contributing to its reduced lifespan [30,31]. Furthermore, the presence of Br was detected, likely captured during calcination, as it is presumed that the muffle furnace was contaminated prior to the analysis.

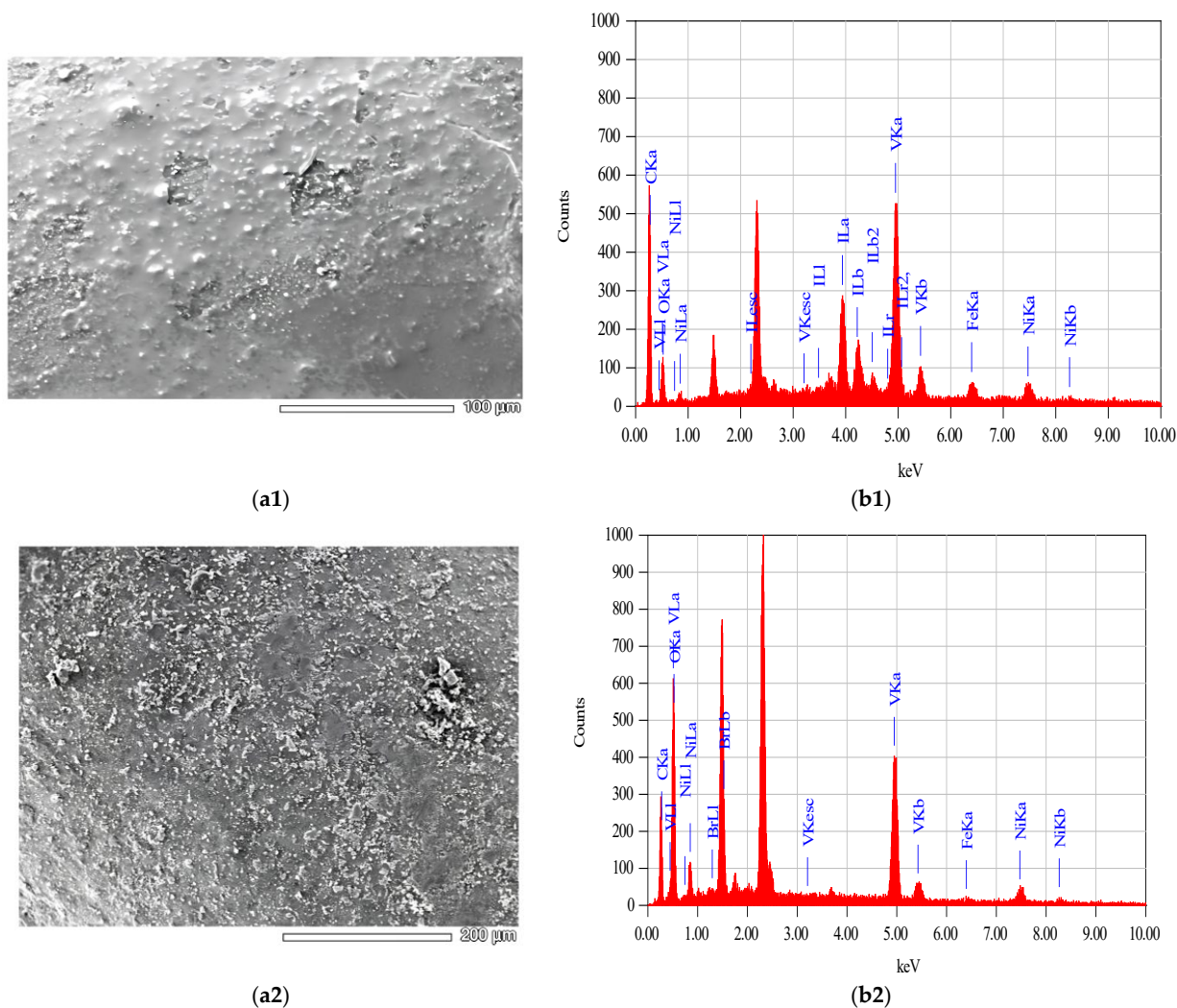


Figure 6. (a1,a2) The morphological analysis of the catalyst before and after the impurity removal process using distilled acetone by SEM. (b1,b2) The EDS spectrum of the catalyst before and after the impurity removal process using distilled acetone.

It is important to note that no significant modifications were observed in the structure of the calcined catalyst granules.

3.4. The Evaluation of the Solids Obtained at the End of Solar Distillation Using XPS and EDS

The solid residue from the distillation was characterized by XPS, where broad spectra ranging from 0 to 1100 eV of binding energy and narrow spectra collected in the high-resolution mode for each element were identified in the sample. The 284 eV shift caused by sample charging was corrected using the peak attributed to adventitious carbon. The broad spectrum (Figure 7) revealed the presence of elements such as Si, Rb, S, O, Cl, N, and C (Figure 8a–d), most of which are related to the clonazepam molecule. It is assumed that Rb originates from pharmaceutical residue. Rb 3d_{5/2}, identified at 108.5 eV of binding energy, is associated with chloride in its chemical environment.

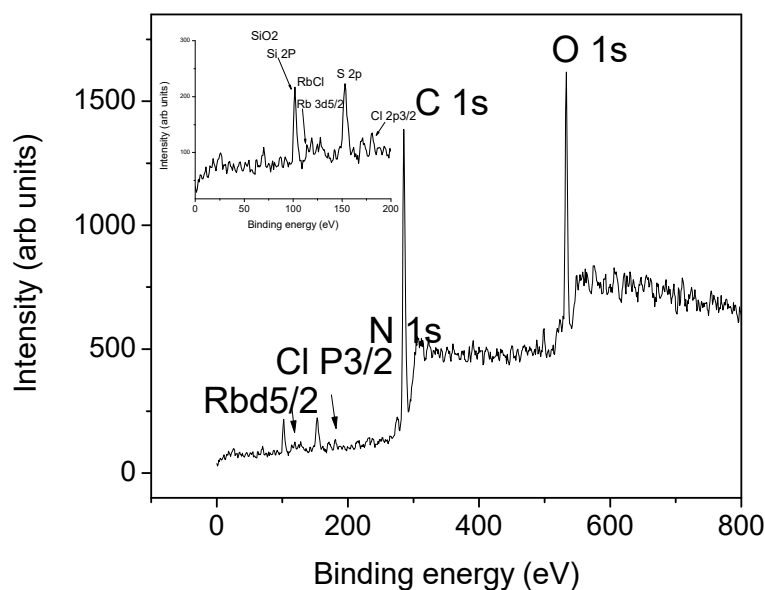


Figure 7. The wide spectrum of the solid from distillation.

The presence of Si 1s is likely due to the glass being attacked during distillation by the acidic fluid handled. In this case, Si was identified as a contaminant. The narrow spectra for each element were processed using Origin 8.1 software. In some cases, each spectrum was deconvoluted to obtain information about the chemical environment of each element and its chemical bonds. The chemical composition of the residual solid was found to include Rb, Si, S, O, Cl, N, and C. The presence of Rb, Cl, N, C, and O is attributed to dissolved organic compounds in pharmaceutical waste liquors. Although Rb is present in a very small atomic proportion, it appears to bind with Cl, as suggested by the XPS doublet observed at 108.5 and 109.9 eV in the narrow spectrum of Rb. Also observed in the same figure, Figure 8, is a doublet that peaks at 111.24 and 112.28 eV, which is attributed to the 3d_{5/2} and 3d_{3/2} signals of Rb⁰.

The Si 2P region (Figure 8b) shows three main peaks located at 103.7, 101.9, and 100.7 eV, which can be assigned to SiO₂, Si-Cl, and Si-C bonds, respectively. These are most likely attributed to the glass being attacked in the acidic medium, presumably due to the presence of chlorine ions in the distillation system originating from the clonazepam drug.

Five peaks were deconvoluted from the C 1s (Figure 8c) region that can be assigned to the organics interacting with O, Cl, and N in the residual material.

The O 1s region (Figure 8d) also shows the O-Si bond and the C-O bond related to the residual clonazepam.

Figure 9 shows the analysis of the energy-dispersive X-ray spectra (EDS) corresponding to the residue from solar distillation, revealing the presence of C, N, Rb, and Cl. Table 3 presents the percentage composition of each element, with C being the most abundant at 63.85%, followed by N and Cl at 18.94% and 3.46%, respectively, and Rb at a much lower value of 0.4%.

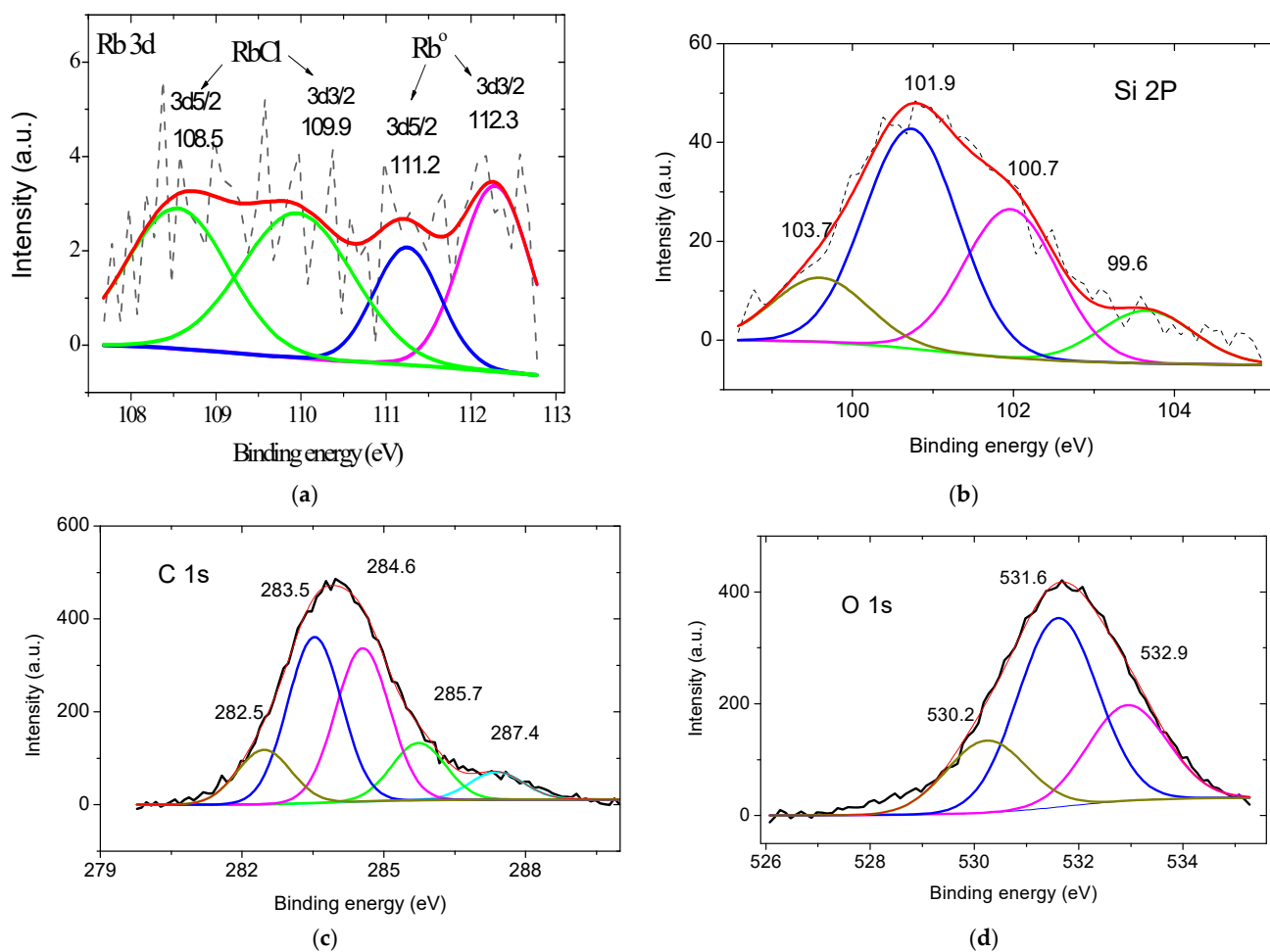


Figure 8. (a) Deconvoluted rubidium, (b) silicon, (c) carbon, and (d) oxygen region spectrums. Note: Different color lines were used in order to get differences between the several binding energies deconvoluted in each spectrum.

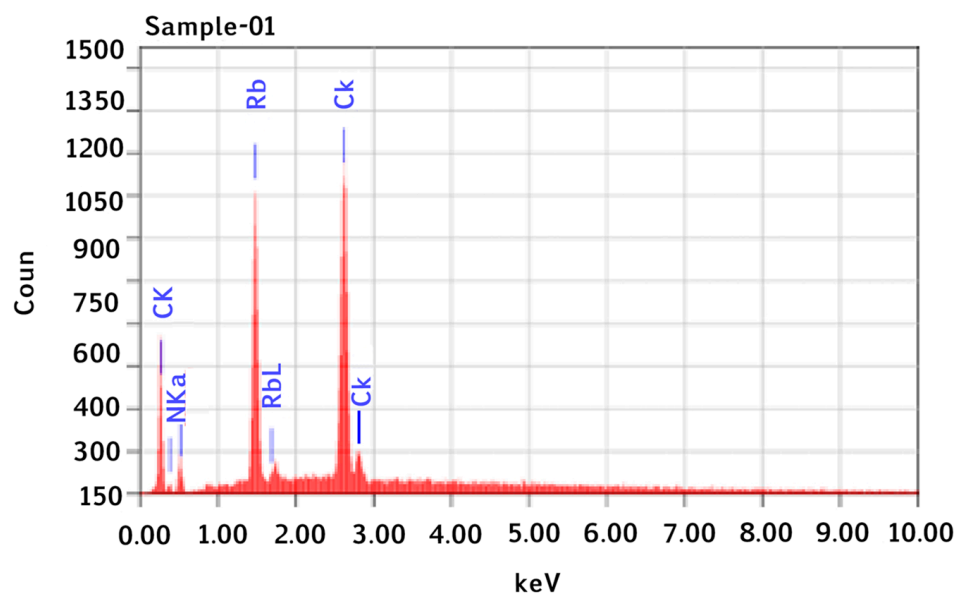
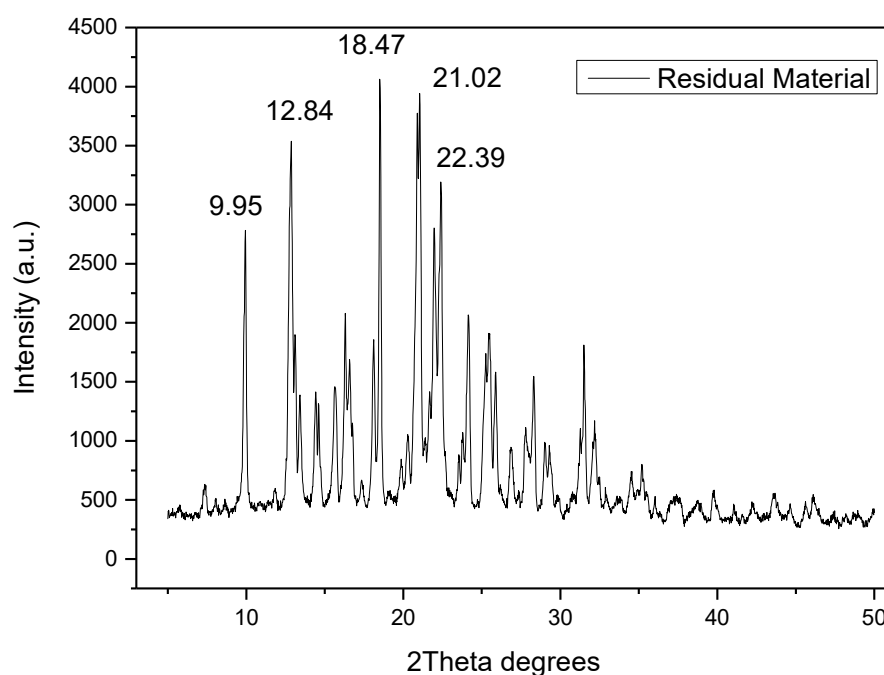


Figure 9. The EDS spectrum of the solid from distillation.

Table 3. The Atomic Composition of the Residue at the End of the Distillation, Obtained from EDS Tests.

Element	Atomic % Cation
C K	63.85
N K	18.94
Cl K	3.46
Rb L	0.40
Total	100.00

In addition, the crystalline phases in the residual material were identified through the diffraction lines (Figure 10), which are associated with SiO₂ (JCPDS 002-0242), primarily from the glass, most likely due to the glass being attacked in the acidic medium during distillation. Other crystalline phases, though in smaller proportions, can also be found in the residual material, originating from the dissolved glass.

**Figure 10.** The XRD analysis of the solid from distillation.

4. Conclusions

In the present study, a single-slope solar still, both with and without solar concentration, was used to recover acetone mixed with clonazepam and other organic compounds from a liquid pharmaceutical residue. The results show that both heating methods achieved efficiencies of 80% and 85%, respectively. Additionally, the distilled acetone demonstrated a purity of 84%, with an optimal separation of clonazepam and other organic compounds present in the original sample.

Furthermore, the reuse of the distilled acetone to clean the granules of a catalyst with high hydrocarbon content, using the calcination method, proved to be effective in removing 29% of the impurities responsible for catalyst poisoning.

Similarly, the solid residues produced during the separation of acetone revealed the presence of rubidium from pharmaceutical waste, which combines with chloride in its chemical environment. Contaminating silicon was also detected, likely due to glass corrosion during distillation. Carbon, chlorine, and nitrogen from clonazepam were also identified.

Therefore, the solar distillation method is effective for recovering solvents for reuse and is considered environmentally friendly and sustainable due to its low-cost production.

Author Contributions: Conceptualization, E.C.T.B. and C.E.B.D.; methodology, E.C.T.B. and C.E.B.D.; validation, L.I.Á.C. and B.A.F.U.; formal analysis, E.C.T.B., L.I.Á.C., C.E.B.D., B.A.F.U. and D.A.S.C.; investigation, E.C.T.B.; data curation, E.C.T.B.; writing—preparation of original draft, E.C.T.B., L.I.Á.C. and C.E.B.D.; writing—review and editing, E.C.T.B., L.I.Á.C. and C.E.B.D.; supervision, L.I.Á.C., C.E.B.D. and B.A.F.U.; project administration, E.C.T.B.; funding acquisition, E.C.T.B. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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