Improvement of PVC Compound Plasticizer Extraction Process*

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ABSTRACT: The Soxhlet method in extracting plasticizers from PVC compounds presents various areas for improvement, such as the amount of solvent used, the time required in the process, and its costs. This study proposes replacing the Soxhlet method with the ultrasound-assisted method to reduce the time, costs, and amount of solvent used in plasticizer extraction. It also proposes using solvent recovered from previous extractions to reduce costs further. The study involved experimentation with three different grades of PVC compounds (medical, cable, and profile) to compare the recovery of plasticizers by the Soxhlet and the ultrasound-assisted methods. The plasticizers obtained were analyzed using gas chromatography, gas chromatography coupled with mass spectroscopy, and Fourier-transform infrared spectroscopy to evaluate the results of each extraction method as compared to reference compounds and determine any possible

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interference. The proposed method yields 96 % of plasticizer extraction, a decrease from 360 to 60 minutes in the extraction process, and a decrease from 150 to 100 ml in solvent use. Complemented by the use of recovered solvent, the proposed method reduces costs from 93,000.00 to 15,000.00 Mexican pesos.

KEYWORDS: extraction / continuous improvement / gases chromatography / gas chromatography coupled with mass spectroscopy

MEJORA DEL PROCESO DE EXTRACCIÓN DE PLASTIFICANTES DE COMPUESTOS DE PVC

RESUMEN: La extracción de plastificantes de compuestos de PVC ofrece variedad de mejoras, como la cantidad de solvente utilizado, el tiempo que toma el proceso y los costos del mismo. Este estudio propone reemplazar el método Soxhlet con el método de extracción asistida por ultrasonido para reducir el tiempo, los costos y la cantidad de solvente utilizado en la extracción de plastificantes. También propone el uso de solvente recuperado de extracciones anteriores para reducir aun más los costos. El estudio supuso la experimentación con compuestos de PVC de tres diferentes grados (médico, cable y perfilería) en la recuperación de plastificantes para comparar los resultados obtenidos por medio del método Soxhlet y por el método asistido por ultrasonido. Los plastificantes obtenidos fueron analizados utilizando cromatografía de gases, cromatografía de gases acoplada con espectroscopía de masas y espectroscopía infrarroja por transformada de Fourier para evaluar los resultados de cada método de extracción al compararlos con la fórmula de los componentes de referencia y determinar alguna posible interferencia. El método propuesto logra una extracción de 96 % de los plastificantes, una reducción de 360 a 60 minutos y de 150 a 100 ml de solvente en el proceso de extracción. Complementado con el uso de solvente recuperado, el método propuesto reduce los costos de 93 000.00 a 15 000.00 pesos mexicanos.

PALABRAS CLAVE: extracción / mejora continua / cromatografía de gases / cromatografía de gases acoplada con espectroscopía de masas

1. INTRODUCTION

Many industries worldwide produce rigid or flexible Polyvinyl Chloride (PVC) compounds for different applications. PVC is one of the most important plastic materials, widely used in construction, packaging, electronics, and daily consumer goods, among other applications.

PVC has good electrical and insulation properties over a wide range of temperatures, excellent durability, and useful life of approximately 40 years. It is also easily processed to obtain the desired specifications of the final product and endures aggressive environments. PVC compounds, produced by different processes such as injection and extrusion, are used for different applications such as construction, cable and wire insulation, door and window frames, ducts and pipes, lining and roofing membranes, wall coverings, floor, tiles, and profiles. PVC compounds also appear in toys such as dolls, bath ducks, inflatable beach toys, wading pools, balls, and some baby care items.

Medical-grade PVC is present in the manufacture of surgical gloves, tubes, serum bags, plasma and blood bags for transfusions and dialysis (studies show that the contact with PVC materials extends the useful life of blood and plasma by 30% (Mariano, 2011).

Being the second most-produced plastic after polyethylene (Jakobi, 2002), PVC manufacturing is immersed in intense competition to improve formulations, whether from the customers' side or the market's side. Reverse Engineering makes it possible to obtain the necessary information to develop new processes to start producing countertype products or products with economic or performance advantages.

A crucial step in the Reverse Engineering of PVC compounds is the solid-liquid separation in the extraction process involving an organic solvent and a solid phase of PVC film. In this process, plasticizers and co-components are identified and quantified (United States Environmental Protection Agency, 2007a).

Many component extraction systems currently include traditional percolation, Soxhlet, and immersion techniques. Moreover, new extraction technologies use critical fluids, microwaves, and ultrasound (Peredo et al., 2009).

Soxhlet extraction has been —and in many cases continues to be— the standard method of extracting solid samples. Since its invention over a hundred and forty years ago, it has been the reference method against which to compare other extraction methods. Significant institutions such as the United States Environmental Protection Agency (EPA) and the Food and Drug Administration (FDA) use this classic technique as an official method for continuous solids extraction. In this procedure, the solid sample is finely pulverized and placed in a porous material cartridge in of Soxhlet extractor's chamber. The extracting solvent, located in the flask, is heated so that its vapors rise to the cooling area and, condensed, start falling, drop by drop, on the cartridge containing the sample, extracting the soluble analytes. When the level of condensed solvent in the chamber reaches the top of the side siphon, the solvent, with dissolved analytes, flows up the siphon and returns to the boiling flask. The process is repeated until all the analytes from the sample have been extracted and are concentrated in the solvent (Universidad Pablo de Olavide, 2004).

Food processing is constantly evolving in response to the challenges and needs of today's society. In this sense, introducing new technologies is vital to reduce processing time, improve operating conditions, and reduce energy needs and environmental costs. The use of ultrasound is an example of a new technology whose application in food processing could improve these parameters. In general terms, ultrasound prominently has importance and application in improving food processes by influencing their kinetics, performance, or the quality of the products (Ulloa et al., 2013).

The use of ultrasound in the extraction of natural products initially aimed to optimize the extraction technique of different matrices of organic nature. Lucena (2019) chose to extract plasticizing compounds present in a synthetic polymeric matrix such as a food-grade PVC film with a perfectly-known composition to study the factors that affect ultrasound-assisted extraction. The incidence of variables such as power, time, and amount of solvent in the extraction was carefully studied.

The incorporation of ultrasound into processes in the food industry is a trend with substantial growth, where the aim is to preserve the sensory quality of food.

The improved efficiency of the extraction of organic compounds by ultrasound is attributed both to acoustic cavitation and mechanical effects. Acoustic cavitation produces the fracture of the cell walls in the plant material, facilitating the penetration of the solvent and allowing the release of the intracellular product. Another mechanical effect caused by ultrasound can also be the agitation of the solvent used for extraction, which increases the surface area of contact between the solvent and the specific compounds of interest, allowing greater penetration of the solvent into the sample matrix (Zhang et al., 2008).

Ultrasound-assisted extraction (UAE) is not a novel extraction method, as it has been widely used in extracting compounds from plants and fruits since the 1980s (Wong-Paz et al., 2017).

A characteristic of high-intensity ultrasonic waves is their ability to act in synergy with other forms of energy, stimulating, accelerating, or improving many processes. This is why several practical ultrasound applications are not exclusively ultrasonic processes but ultrasonically assisted processes. Such a situation is particularly important in those processes related to the food industry, where the application of ultrasonic waves requires clean energy (Knorr et al., 2004, Chemat et al., 2011; Mason et al., 1996).

Sonication generates the formation and collapse of microscopic bubbles that release large amounts of energy in the form of heat, pressure, and mechanical stress (Briones-Labarca et al., 2015); in this way, microturbulence and increased diffusion are generated (Shirsath et al., 2017)

Efforts to avoid the disadvantages of conventional extraction methods have resulted in the implementation of other techniques known as "green technologies". In addition to presenting various advantages in the extraction process, these technologies do not negatively impact the environment and reduce the use of organic or green solvents significantly (Dar et al., 2015).

The main environmentally-friendly techniques are ultrasound (UAE), microwave (EAM), pressurized liquids (ELP), and supercritical fluids-assisted extraction (Carciochi et al., 2017). UAE and EAM are the most used because of their high yields of bioactive compounds, smaller amounts of solvent used, and shorter extraction times (Bandar et al., 2013). However, despite having these alternatives, few investigations use these techniques to extract and recover bioactive compounds from citrus fruit residues (Khan et al., 2010; Boudhrioua et al., 2016).

Currently, the use of ultrasound in food processing has increased due to its advantages over conventional processes in terms of time and temperature, effective mixing, increase in mass and energy transfer, and reduction of thermal gradients. Concentration, selective extraction, faster response to extraction process control, increased production rate, and removal of microorganisms and enzymes without destroying food nutrients are also advantages of using ultrasound (Campo et al., 2018).

Spinella et al. (2015) evaluated the effect of different variables (temperature, power, moisture content of the grain) during ultrasound-assisted solvent extraction of high stearic sunflower oil. The process allowed a 92% increase in the oil yield in the first thirty minutes compared to the conventional Soxhlet method.

Corona et al. (2016) also mention that the ultrasound-assisted process showed a greater yield than conventional methods in extracting phenolic compounds from chia seeds.

There are two methods for ultrasound-assisted extraction: directly, through a system of probes immersed in the liquid-solid system with very high intensities or, indirectly, using an ultrasonic water bath and placing the sample in a flask with the solvent, so the waves tavel through the water to the plant material. These techniques are used to obtain polysaccharides such as pectins, essential oils, bioactive compounds, and proteins (Bromberger et al., 2018).

These references guided the authors to study the substitution of the conventional Soxhlet method used in the Reverse Engineering of the Research and Development Department of a PVC compounds company located in the South of Tamaulipas, Mexico by the ultrasound method. The conventional method wastes person-hours and usually delays delivery of results, and when it uses a certain amount of flammable solvent, it compromises the safety of the staff. This study aims to improve safety conditions by reducing the person-hours invested in the PVC compounds extraction process and minimizing the exposure time of the personnel to flammable solvents, thus diminishing the process' environmental and economic impact.

2. METHODOLOGY

The research for this paper was carried out in the Product Design and Development Laboratory of a leading manufacturer of PVC compounds in Latin America, located in the South of Tamaulipas, Mexico. The extraction process of plasticizers from PVC compounds uses flammable solvents. Safety measures implemented due to incidents that have occurred a couple of times cause delays in delivering results. In order to reduce exposure risks and delays, this study searched for improvement opportunities related to the time and cost of the solid-liquid extraction process currently carried out using the conventional Soxhlet method (United States Environmental Protection Agency, 1996). Comparative data on solvent consumption was collected over a period of twelve months, from January to December 2019. Likewise, with the use of appropriate techniques and methods, a proposal for ultrasound-assisted extraction technology (UAE) was analyzed and evaluated. Its efficiency and optimal conditions were designed for this new system.

This section details the variables used in the proposed UAE method (United States Environmental Protection Agency, 2007b.) to obtain the liquid extracts of three flexible PVC compounds for medical, profile, and cable applications.

Table 1

Experimentation test variables

Solvent amount (milliliters)	Extraction time (minutes)
50	30
50	30

2.1 Equipment, Materials and Services

The study used different equipment and instruments: a Mettler Electronics ultrasound bath equipment (model ME-4.6) of 85 watts of power and 50/60 Hz frequency, which served for the development of the UAE method tests. Special glass equipment was used for extraction by the Soxhlet method. Two grams of PVC compound were pressed using a Collin brand hot plate press and a Dake Corporation cold plate press in order to maximize the contact area between compound and solvent for both extraction methods. In one of the final stages of the plasticizer extraction process, a Lab-line brand vacuum oven was used to eliminate the remaining solvent. Analytical balances of the Sartorius brand, chronometers, and thermometers were used, among other essential instruments.

We also used equipment for plasticizer extract analysis, such as Perkin Elmer Infrared Spectrophotometer Spectrum 100 model, Agilent Technologies 7890 °C Gas Chromatograph (Column 19095Z-423 of 300 °C: 3μm x 530μm), a Clarus 680 model gas chromatograph coupled to a Perkin Elmer Clarus SQ 8T mass spectrometer model (Elite 5 MS Column Part N.º. 9316282 m dimension of 30 m. ID 0.25, DF 0.25 temperature range from -60 °C to 350 °C).

2.2 Research Design

This study used commercial compounds of medical-grade, cable, and footwear PVC produced by the company in South of Tamaulipas to extract plasticizers by the Soxhlet and the ultrasound-assisted methods. The Soxhlet method, one of the most widely used for extracting solid samples, serves as a standard against which other methods can be compared. The Soxhlet method obtains high levels of analytes by keeping the sample in contact with fresh solvent during the process; the operating methodology is simple, and complex purification processes are not required. However, this technique is costly and requires investing considerable time in monitoring.

The objective of having three different PVC compounds was to compare their plasticizer content to determine their recovery percentage according to their formula and the application of the alternative method in the Reverse Engineering process. Each formulation was carried out by five replicates for each method. The recovery results obtained were used to determine the reliability of each method.

The samples submitted for analysis were weighed before the extraction process. Two grams of each compound were pressed to form a very thin film to allow a greater contact area between the solid and the solvent. The process involved three different stages: extraction of the plasticizer, distillation of the anhydrous ethyl ether solvent, and drying of the plasticizer in a vacuum for both methods. We explain the process stages for both methods in the following sections of this article.

2.3 Soxhlet Method

We extracted the plasticizers from the three PVC compounds using the Soxhlet method. The procedure was carried out as follows:

1. Place 2 grams of PVC compound film in the Soxhlet system.
2. Divide 150 ml. of solvent between a flat-bottomed flask and a Soxhlet system, fit together and place them in a water bath at a temperature of 35 +/- 5 °C for 6 hours.

3. Distill the solvent with the extract for one hour at a temperature of 55 °C. The volatile solvent condenses in a receiving flask. Then it can be stored for later use.
4. Once the distilling is over, remove traces of the solvent by placing the flat-bottomed flask in a vacuum oven for one hour at a temperature of 65 °C.

2.4 Ultrasound-assisted extraction method

A Mettler model ME-4.6 ultrasound bath equipment was used for the ultrasound-assisted plasticizer extraction. This type of equipment is usually used for cleaning medical instruments, metal parts, and glassware. Given its low power (85 watts) and frequency (50/60 Hz.) it is not the ideal equipment for a study like this, however. Knowing these limitations, we proceed as follows:

1. Install the ultrasonic bath and fill it with 800 ml. of tap water at room temperature.
2. Put 2 grams of PVC compound film and 50 ml. of solvent recovered in an Erlenmeyer flask and sonicate for thirty minutes. Once this time has elapsed, transfer the solvent with the extract to a flat-bottomed flask.
3. Add 50 ml of recovered solvent to the Erlenmeyer flask for another thirty minutes and then transfer to the same flat-bottomed flask mentioned in step 2.
4. Repeat steps three and four of the Soxhlet method.

3. RESULTS

The company uses anhydrous ethyl ether as a solvent for extracting plasticizers from PVC compounds. The use of this solvent entails considerable expense, so to reduce costs, this study used solvent recovered from previous extractions as a substitute for pure solvent. The study used gas chromatography and mass spectrometry to analyze the recovered solvent to ensure its purity. After the purity of the solvent was determined, the three different grade PVC compounds were submitted to the extraction process using the techniques mentioned above. The study used infrared spectroscopy (FTIR) and gas chromatography (GC) to analyze the plasticizer extracts and compare them to the plasticizer reference for each compound.

3.1 Recovered solvent effect analysis

During the last ten years, the company where the study was carried out has been using 99.99 % pure anhydrous ethyl ether as a solvent. The use of this solvent entails problems and concerns since it is highly flammable and expensive. For this reason, this study seeks to reduce the amount of solvent used by reusing it in future extractions. For the said reuse of the recovered solvent, it was necessary to carry out chromatographic analysis using gas chromatography and gas chromatography coupled to mass spectrometry to determine the absence of components or residues of past extractions in the solvent.

3.2 Solvent analysis by gas chromatography

Gas chromatography is the scientific method used to confirm the presence or absence of a compound in a given sample. When we see a substance at first glance, it seems that all of it is made up of the same material. However, this is not necessarily the case. We can precisely determine the type of elements that make this substance up and the quantity of each in a given sample with this type of scientific analysis. Figure 1 shows the chromatograms of the pure and recovered anhydrous ethyl ether. The similarity between them stands out: the results showed that the pure anhydrous ethyl ether had 99.92 % purity, while the recovered solvent had a 99.91% purity. Approximately at the fourteenth minute, the recovered solvent chromatogram showed a small signal, equivalent to 0.00026% of the solvent. For this reason, the study deemed it as not significant.

Figure 1

Comparison of Pure Anhydrous Ethyl Ether vs Recovered by Gas Chromatography

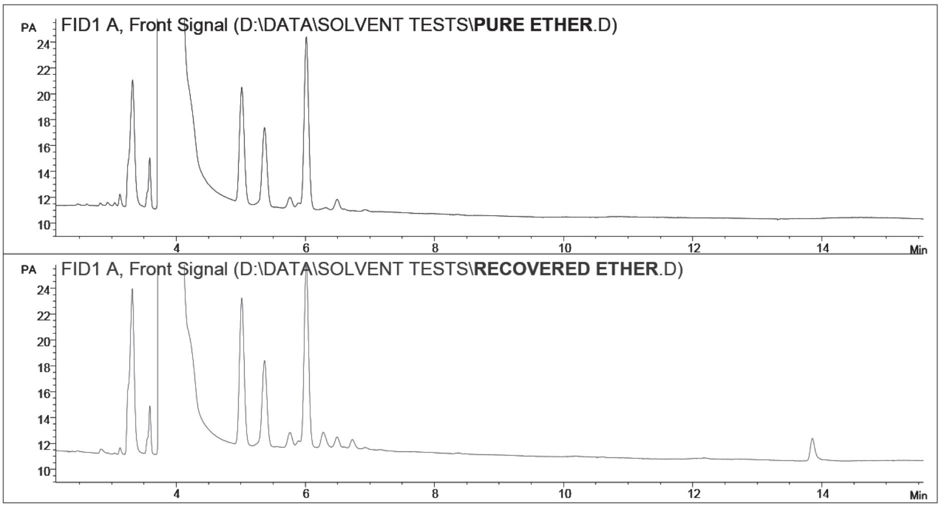
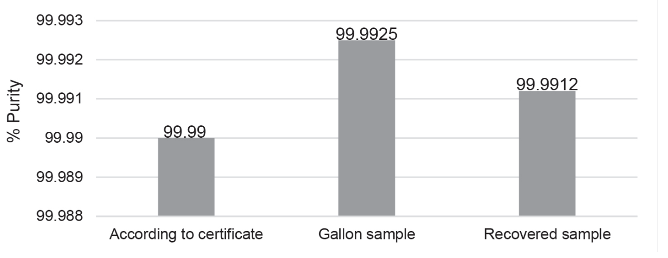


Figure 2 shows the purity of the pure and the recovered samples of solvent obtained by chromatography. Both of them have higher percentages of purity than that established by certification. This leads to the conclusion that both solvents comply with the specification of 99.99 % purity.

Figure 2

Comparison Pure Anhydrous Ethyl Ether vs Anhydrous Ethyl Ether Recovered by Chromatography



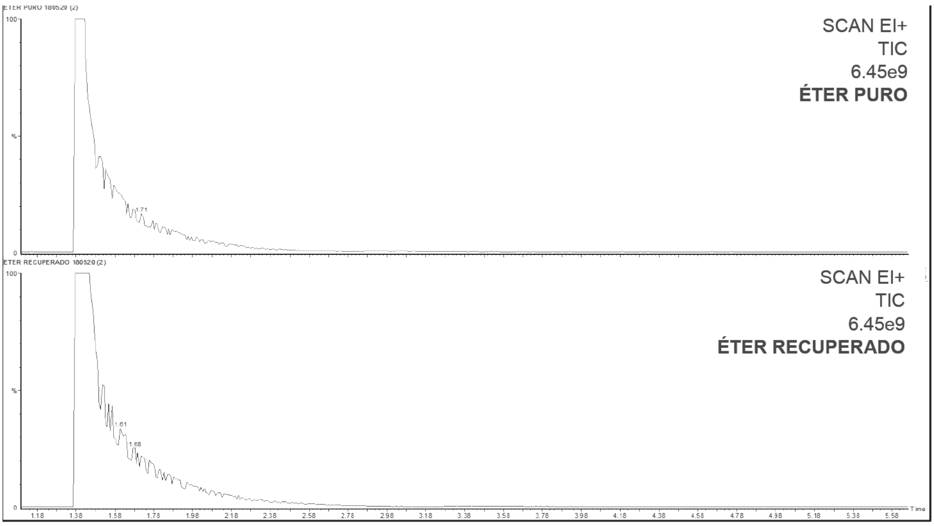
3.3 Solvent analysis by Gas Chromatography coupled to Mass Spectrometry

Gas chromatography coupled with mass spectrometry (GC/MS) enables the separation, identification, and quantification of mixtures of volatile and semi-volatile substances. The separation of these substances depends on the different distribution of the substances studied between the mobile and stationary phases that make up the system. Once the substances are separated, they are fragmented, and their fragmentation pattern is analyzed and compared with information contained in a mass spectra database for their preliminary identification. Each substance's definitive identification and quantification is made using a reference substance (Universidad Veracruzana, 2019).

The GC-MS analysis of the pure and recovered solvent ensured the recovered solvent would not affect the efficiency of the plasticizer extractions. Figure 3 shows that there are no significant differences between the chromatograms of the pure and recovered solvents

Figure 3

Comparative chromatogram of pure Anhydrous Ethyl Ether vs Recovered Anhydrous Ethyl Ether obtained by Mass Spectrophotometer



3.4 Extraction of plasticizers by methods

The study consisted of five extractions of plasticizers from each of the three different grade PVC compounds, using the two methods mentioned before and comparing the actual and obtained content of plasticizers, as shown in Table 2.

Table 2

Results of Experimentation of Soxhlet and EAU Extraction methods

Degree type	Actual content of plasticizers by compound formula (%)	Plasticizer obtained by Soxhlet Extraction (%)	Plasticizer obtained by Ultrasound Assisted Extraction (UAE) (%)
Medical	43.68	43.26 Recovered: 99.04	42.34 Recovered: 96.94
Cable	20.72	21.23 Recovered: 102.43	19.96 Recovered: 96.30
Profiles for Windows	36.28	37.33 Recovered: 102.88	36.54 Recovered: 100.71

We determined the amount of extracted plasticizer in grams, measuring the initial and final weights of the receiving flask of both extraction systems (Equation 1). We then calculated the content of extracted plasticizer as a percentage (Equation 2) and, finally, used Equation 3 to determine the amount of extracted plasticizer vs. the amount contained in each formula.

Equation 1:

$$\textit{Pounds of recovered plasticizer} = \text{final flask weight} - \text{initial flask weight} \tag{1}$$

Equation 2:

$$Percentage\ of\ recovered\ plasticizer = \frac{\text{pounds of recovered plasticizer x 100}}{initial\ weight\ of\ composite\ film} \tag{2}$$

Equation 3:

$$Plasticizer\ recovery\ percentage\ obtained\ from\ real\ formula\ = \tag{3}$$

$$\frac{percentage\ of\ plasticizer\ obtained\ x\ 100}{Actual\ content\ of\ plasticizer\ in\ compound\ formulation}$$

These results show that the UAE method did not extract all the plasticizers contained in the PVC compounds. In contrast, the Soxhlet method extracted all the plasticizers and an additional part of the liquid raw materials present in the compound formulae. Results are a consequence of the differences between the different compound grades. It is worth mentioning that each raw material has particular characteristics, and some show greater or lesser migration than others.

3.5 Analysis of Plasticizer Extracts by Analytical Equipment

3.5.1 Analysis of plasticizer extracts by Infrared Spectroscopy

We compared the plasticizer extracts of the medical (Figure 4), cable (Figure 5), and profile (Figure 6) grades of the PVC compounds obtained by both methods using infrared spectrometer equipment. When we compared the extracts obtained by both methods with the reference plasticizer of the compound formula, it was evident that there were no differences between them in the infrared spectra.

Figure 4

Comparison of liquid extracts of medical compound by FTIR

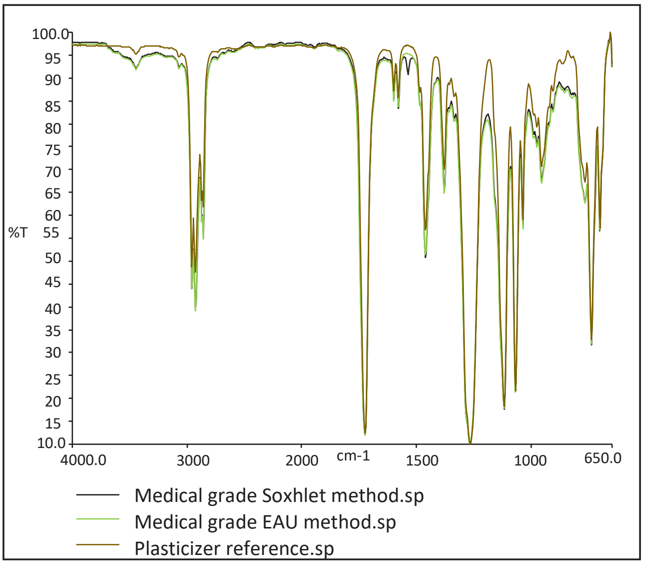


Figure 5

Comparison of liquid extracts of cable compound by FTIR

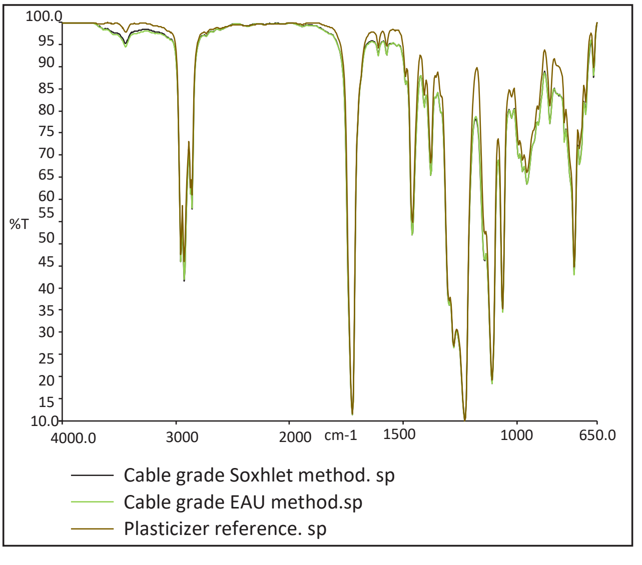
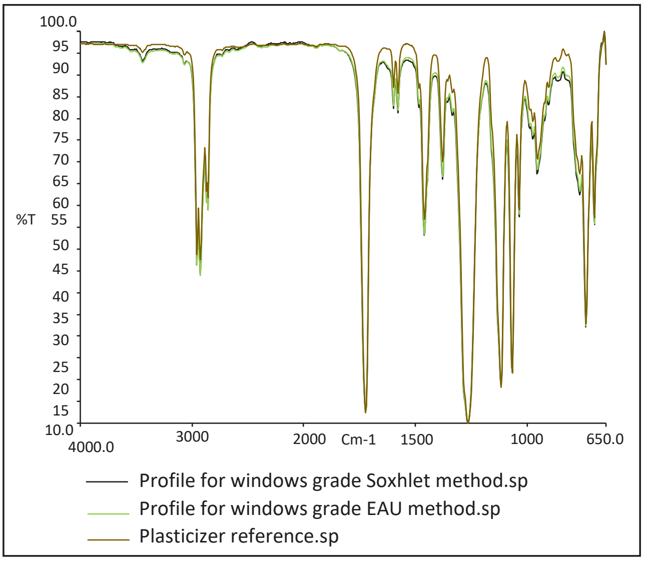


Figure 6

Comparison of liquid extracts of profile for windows grade compound by FTIR



For these analyses, we used a Perkin Elmer Fourier transform infrared spectrophotometer Spectrum 100 model at 16 scans in a range of 4,000 to 650 cm-1 with a resolution of 4.0 cm-1 using as a sampler a Pike Technologies ATR model MIRacle-ATR. The equipment had a certified calibration on February 19, 2020, valid for six months. The results correspond exclusively to the analyzed samples.

3.5.2 Analysis of plasticizer extracts by gas chromatography

We also analyzed the plasticizer extracts of the medical (Figure 7), cable (Figure 8), and profile (Figure 9) grades of the PVC compounds obtained by both methods using gas chromatography. The equipment used was an Agilent Technologies 7890°C with a 19095Z-423 column (300°C: 3m x 530 µm x 3 µm), which had a qualified calibration since November 21, 2019, with an expiration date of twelve months.

The chromatograms of the extracts show that there were no abnormal signals in the plasticizer obtained from the extraction process nor signals or traces of the use of the recovered solvent compared to the reference compound.

Figure 7

Comparative chromatogram of medical grade extracts vs plasticizer reference by Gas Chromatography

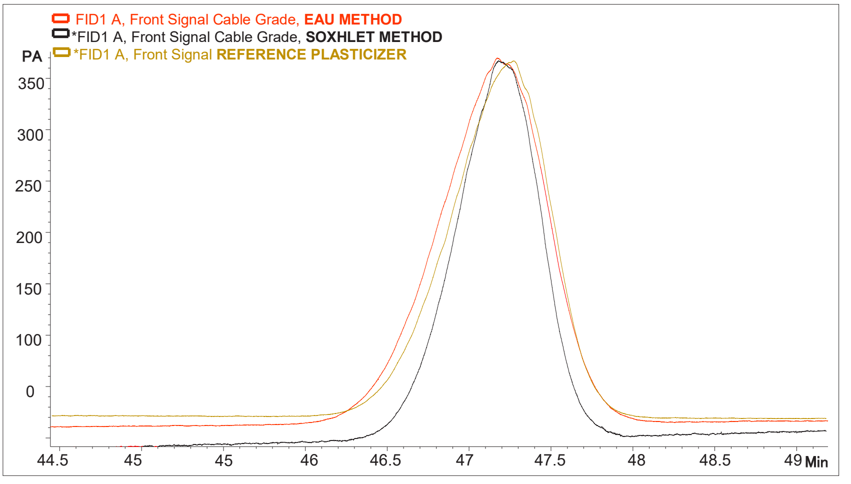


Figure 8

Comparative chromatogram of cable grade extracts vs plasticizer reference by Gas Chromatography

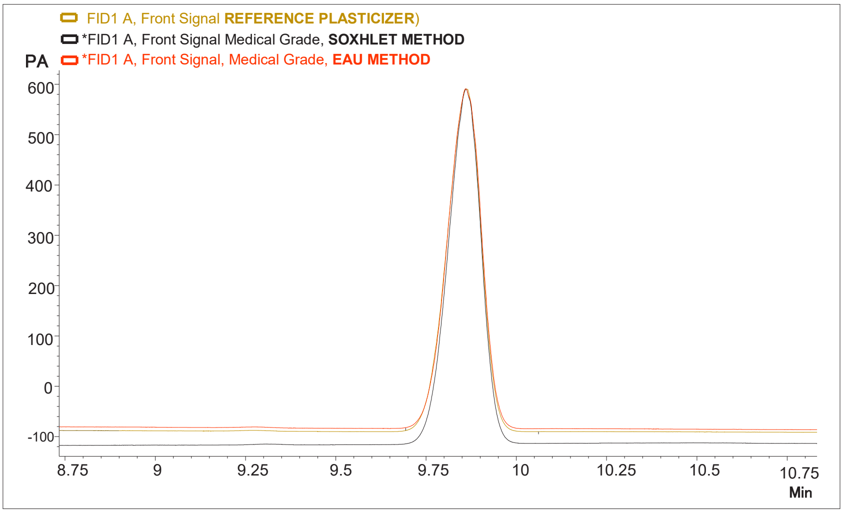
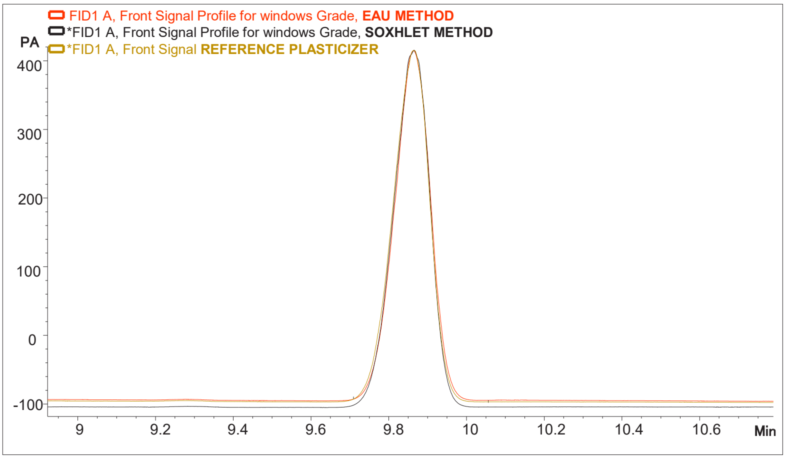


Figure 9

Comparative chromatogram of profile for windows grade extracts vs plasticizer reference by Gas Chromatography



3.6 Economic Impact Analysis

An economic impact analysis was also part of the study to determine the cost of each plasticizer extraction for both techniques. This part of the study considered the number of extraction processes carried out during 2019 as a base for an annual projection of the costs. In the case of the UAE method, 100 milliliters of solvent were used for each extraction process which took sixty minutes. In the case of the Soxhlet method, 150 milliliters of solvent were used for each extraction process which took three hundred and sixty minutes. For both techniques, the cost per gallon (3.78 liters) of pure anhydrous ethyl ether solvent was estimated at $1656.00 Mexican pesos. Table 3 shows comparisons for both methods.

Table 3

Cost Comparison of Extraction Methods

	Soxhlet Extraction	Ultrasound Assisted Extraction
Milliliters of solvent used	150 ml	100 ml
Total, used solvent	23.25 liters	15.5 liters
Total solvent cost	$10,510.00	$6780.00
Invested worker days	117 days	20 days
Total labor	$93,000	$15,500.00
Total from solvent confinement	$9,229.06	$9,219.38
Total annual cost (with pure solvent)	$112,739.26	$31,500.15
Total annual cost (with solvent recovered)	$93,000.00	$15,500.00

During 2019 the company carried out 155 extractions using the Soxhlet method with an annual expense of $112,739.26. With the use of recovered solvent for the same number of extractions, the annual cost would decrease to $93,000.00. If the EAU method were to be used in the same number of extractions, the annual cost would be $31,500.15. With the use of recovered solvent for this method, the annual cost would be even lower: $15,500.00. These results signal the following benefits:

- The cost of pure solvent purchase per sample decreased by 64.52%.
- The cost of labor costs decreased by 83.3%.
- The total costs decrease by 72.05% by replacing the Soxhlet extraction method with de UAE method.
- Finally, the total cost of extraction decreased by 83.33% by using recovered solvent in the ultrasound-assisted extraction method.

4. CONCLUSIONS

- As a result of this study, we propose to replace the current Soxhlet extraction method with the ultrasound-assisted extraction method, both described by the Environmental Protection Agency.
- Extracting plasticizers from flexible PVC compounds using the UAE method yields a reduction of five hours per extraction and $81,239.11 (Mexican pesos) in the annual cost (i.e., a decrease of 78.4%.).
- For optimal plasticizer extraction using the UAE method, sonicate 50 ml of recovered solvent in a flask for 30 minutes. Remove the solvent and add another 50 ml of recovered solvent and sonicate for another 30 minutes.
- Compared to the current Soxhlet method, the UAE method offers similar amounts of plasticizer extraction, decreased process time, more effortless procedure, and versatile and reliable technology.
- Both extraction methods have good results; the difference lies in the ease, time, and costs involved in each method involves.
- The applicability of the proposed UAE method with solvent recovered by Gas Chromatography and Gas Chromatography coupled to Mass Spectrometry has been demonstrated, which would mean an 83.3% reduction in solvent costs.
- There is variability in the plasticizer recovery percentages of both methods. This is probably caused by the compatibilities of the plasticizer with the other raw materials that make up the compounds.

- The results obtained encourage future research for which we recommend the use of more powerful sonication equipment (200 W and 20 or 25 kHz).

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