SEM, AFM and CLSM microscopic techniques as tools for the characterization of cellulose, polyaluminum and aluminum recovered from Tetra Pak packaging

C. Barrera-Díaz[1], L.I. Ávila Córdoba [2], F. Ureña-Núñez[3], G. Martínez-Barrera[4], V. Varela-Guerrero[1] and L. Rosales-Hernández [2]

[1] Centro Conjunto de Investigación en Química Sustentable UAEM-UNAM. Universidad Autónoma del Estado de México Campus El Rosedal. Autopista Ixtlahuaca-Atlacomulco, km 14.5, C.P. 50200. Estado de México.
[2] Unidad Académica Profesional Tianguistenco, Universidad Autónoma del Estado de México, Paraje El Tejocote s/n, San Pedro Tlaltizapán, 52640, Tianguistenco, Estado de México.
[3] Instituto Nacional de Investigaciones Nucleares, Carretera México-Toluca s/n, 52750, La Marquesa Ocoyoacac, Estado de México.
[4] Laboratorio de Investigación y Desarrollo de Materiales Avanzados (LIDMA), Facultad de Química, Universidad Autónoma del Estado de México, Km.12 de la carretera Toluca-Atlacomulco, San Cayetano 50200, México.

Nowadays, the industry requires materials with very specific properties, for example, low molecular weight, high chemical and/or mechanical resistance, impermeable to various types of substances and durability. The combination of such characteristic results in composite materials such as Tetra Pak®[1]. However, inadequate use and improper disposal of this type of packaging, leads to major pollution problems, so the use of different separation and recycling processes become relevant in minimizing the environmental impact [2].

In this chapter, the characterization of the morphological features of sub-products of Tetra Pak packaging using SEM, AFM and CLSM microscopic techniques is presented. In a first step, the separation of the components of the Tetra Pak was done using hydropulping mechanical process; the principal obtained products include cellulose, aluminium and polyethylene+aluminum. The last one called Polyaluminum. After this, the morphological analysis was obtained. The results show that such microscopy techniques are adequate for describe the high degree of purity of the components after recycling.

Keywords: Scanning Electron Microscopy; Atomic Force Microscopy; Confocal Laser Scanning Microscopy; Tetra Pak packaging; Polyaluminum; Mechanical Recycling.

1. Tetra Pak packaging: Current issues and recycling

Globally, serious environmental problems are caused by the generation and accumulation of solid waste, particularly municipal solid waste, which is generated in private homes, shops, offices and services, and those who are not classified as hazardous wastes [3, 4]. Municipal solid waste consist of a mixture of various waste products, many of them can be recycled such as glass, ceramic, metal, plastic, multilayer packaging (Tetra Pak®), paper, cardboard among others.

At the moment, the recovery of solid waste has established itself as a major pillar of sustainability, as it maximizes the economic value through savings of energy and materials, as well as minimizes environmental and social impact. Recycling means less waste generation, better layout and optimization of the operation of transfer stations and landfills as well as in the demand reduction for resources.

Tetra Pak containers are made with cellulose (75%), polyethylene (20%) and aluminum (5%), which can be recovered by recycling the containers using different techniques. Figure 1 shows the six different layers of the aseptic package.

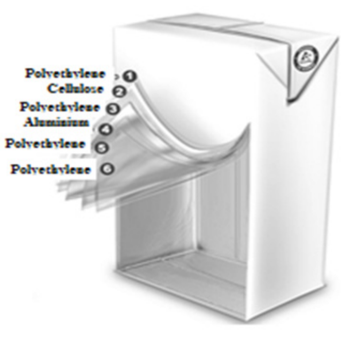

Fig. 1. Layers of Polyethylene, Cellulose and Aluminum in a Tetra Pack packaging [5].

In Mexico, the recycling rate of this type of packaging has increased significantly: in 2003, the percentage was 0.9%, which increased to 10.8% in 2011 and 19.5% in 2013 [6].

Recycling Tetra Pak® packaging is based on the hydropulped process, where the separation of two components is achieved, cellulose and polyethylene+aluminum. Cellulose is used to produce recycled paper and polyethylene+aluminum is used as raw material in the manufacture of corrugated sheets and building panels, as is the Tectan, an agglomerated material wood, who is used as substitute for furniture [7, 8].

The economic impact that represents the disposal of the various constituents of the multilayer container is attractive only if it is possible to recover materials in large quantities, for example, polyethylene+aluminum represents approximately 25% of recycled material. Among the processes of separation of aluminum there are: incineration (for the production of aluminum oxide in the cement industry), pyrolysis and use of thermal plasma [5, 9, 10].

It must be mentioned that each of the technologies mentioned above involves changes in the properties of separated materials (cellulose, polyethylene+aluminum and aluminum), from the multilayer container, which can affect post-consumer uses. Characterization of the waste materials using microscopic techniques as Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Confocal Laser Scanning Microscopy (CLSM) allows to observe changes in the physicochemical properties and morphology.

2. Characterization of cellulose, polyaluminum and aluminum by microscopic techniques

2.1 Materials

In this work, Tetra Pak® of the same commercial brand was used. Sample analysis was carried out by Scanning Electron Microscopy with a JEOL microscope model JSM 6010LA, Atomic Force Microscopy with a VEECO microscope model CP-II: SPM Digital, and Microscopy Confocal Laser Scanning with a model LEICA TCS SPE / CTR 4000 device.

2.2 Experimental procedure

Tetra Pak® collection was carried out to gather post-consumer packaging of the same commercial brand. Subsequently the beverage containers (without plastic screw-on cap), were compacted and cut lengthwise to have an average size of 28 cm x 19 cm (edges were discarded), and then washed. Such process is shown in Figure 2.

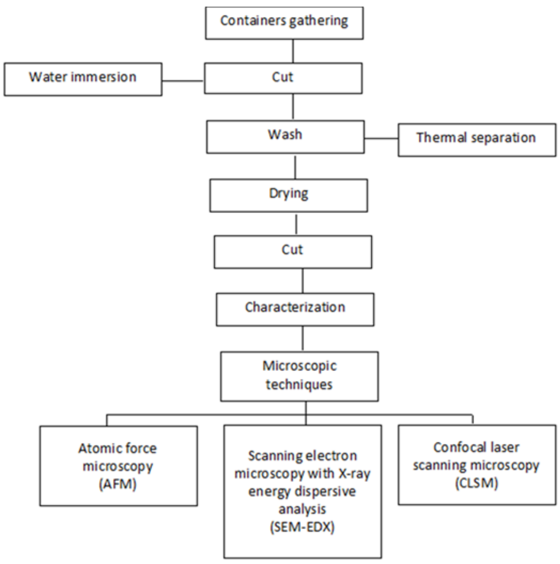

Fig. 2. Schematic diagram of the procedures carried out for the experimental section.

After this, the samples were placed in a tray to clean the inside and outside of the containers by using soap and a soft sponge, removing any residue. After the cleaning of the containers, the process to separate cellulose and polyethylene+aluminum was carried out through the hydropulped technique (submersion in water and stirring for 48 h). Cellulose was mechanically separated.

Polyethylene+aluminum samples were dried by direct exposure to the sunlight for about three hours. Recovery of aluminum from Tetra Pak® and polyethylene+aluminum, was by pyrolysis of multilayer containers.

Characterization of the recovered materials was performed by morphological analysis and composition. To assess structural and/or functional changes, particle size and chemical composition, the techniques of scanning electron microscopy coupled to a X-ray scattering analyzer, (SEM-EDS operated on secondary electrons mode), atomic force microscopy (AFM-tapping mode) and Confocal Laser Scanning Microscopy (CLSM) were used.

The samples were analyzed without any special preparation, they were cut to a size of 3 mm x 3 mm ensuring that were perfectly clean and free of grease to prevent surface contamination. In the same way the perfect adhesion to the support and vibration isolation was careful. Technical specifications for AFM analysis are shown in Table 1.

Table 1. Technical specifications for AFM characterization of recovered Tetra Pak®, polyaluminum and aluminum.

Parameter	**Tetra Pak®**	**Polyaluminum**	**Recovered Aluminum**	**Aluminum Can**
Scanning lines	448	448	448	448
Scanning points	448	448	448	448
Scanning velocity	0.28 Hz	0.27 Hz	0.28 Hz	0.28 Hz
Scanning area	60 µm	90 µm	5 µm	90 µm

3. Results

3.1 Tetra Pak packaging

3.1.1 Scanning Electron Microscopy (SEM)

Fig. 3 shows the morphology of Tetra Pak® where five of the six layers of the containers is observed. The layers identified in the images obtained by SEM at 100X, correspond to: (1) LDPE, (2) cellulose, (3) LDPE, (4) aluminum, and (5) LDPE. As can be seen between the second and third layers, the plastic adheres to the paperboard. It is noteworthy that during its preparation, polyethylene is introduced into the cellulose fibers at a temperature of 106 °C. Upon cooling, it also binds to aluminum acquiring solid state. At a higher resolution, 250X, four layers are identified as: (1) cellulose, (2) LDPE, (3) aluminum, and (4) LDPE.

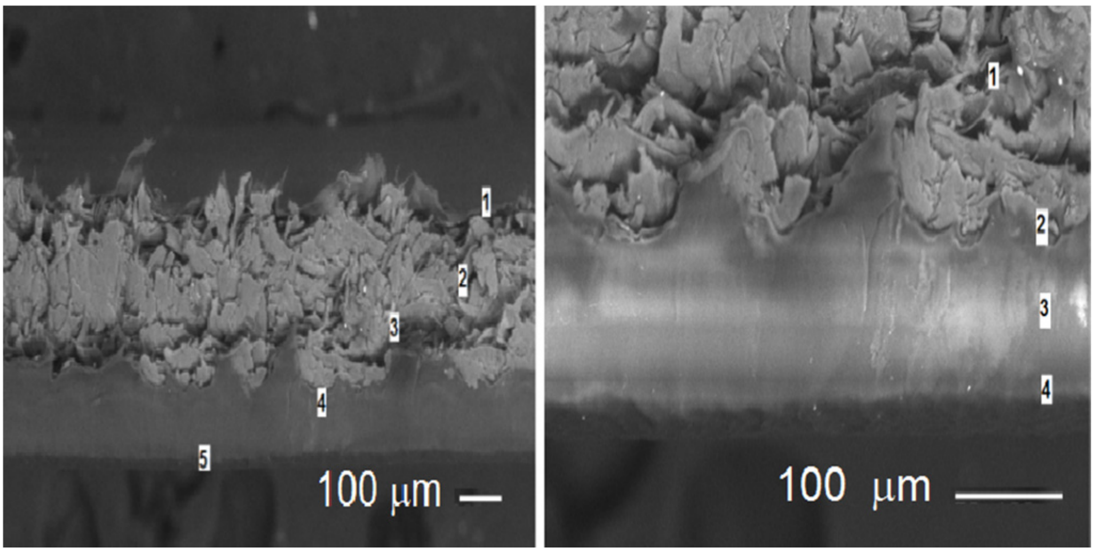

Fig. 3. SEM images of Tetra Pak® waste.

3.1.2 Atomic Force Microscopy (AFM)

The topological characterization of Tetra Pak packaging by AFM is shown in Figure 4. Aluminum characteristic lines are observed in the four images. The blue area is at the lowest height, between 1.3 and 1.65 µm. In this case of the aluminum peaks, this reach heights from 7.60 to 10.4 microns. In Figure 4 (c), it is possible to observe non-uniformity in the sample.

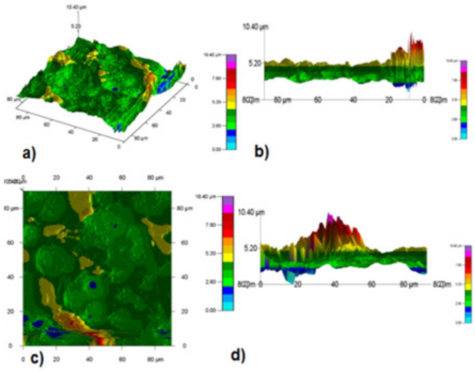

Fig. 4. AFM images of different sections of Tetra Pak packaging: (a) Isometric; (b) Side; (c) Top; (d) Frontal.

3.1.3 Confocal Laser Scanning Microscopy (CLSM)

Confocal laser scanning microscopy (CLSM) has the significant advantage of permitting recovery monitoring at a defined image plane of the Tetra Pak packaging specimens. In the comparative images for Tetra Pak® samples (Figure 5), the fluorescence can be identified with changes in intensity and color. Absorption occurs in three wavelengths: blue (405 nm), green (488 nm) and red (532 nm). Moreover, in Figure 5a, 5b, 5c and 5d, little fluorescence is observed because the analyzed specimen is a composite material.

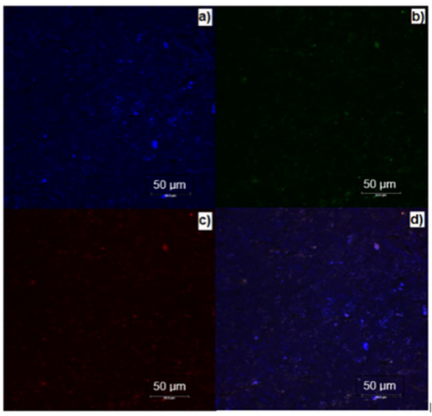

Fig. 5. CLSM images of Tetra Pak® packaging.

3.2 Cellulose

3.2.1 Scanning Electron Microscopy (SEM)

Figure 6 shows images of recovered cellulose obtained by Scanning Electron Microscopy (SEM). Smooth and homogeneous surface as well as dispersed particles are observed. Such particles with diameters lower than 5 µm. Moreover, some channels produced by the presence of aluminum materials, give the appearance of cracks. Some dark regions on the cellulose show rough surfaces (indicated by circles). Moreover, a few pores are observed (black points).

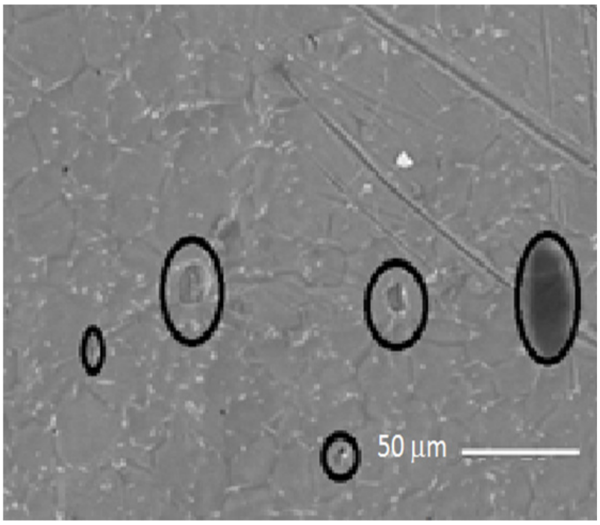

Fig. 6. SEM image of recovered cellulose from Tetra Pak packaging.

3.2.2. Confocal Laser Scanning Microscopy (CLSM).

Different aspects are observed for recovered cellulose of Tetra Pak packaging, which including well-defined cellulose microfibrils with homogeneous surface and diameters ranging from 10 µm to 40 µm. The cellulose showed brighter color and more intensive contrast (Figure 7a), than those for gray contrast. The array microfibril structure and uniformity of the surface is evident. Moreover, cellulose has high degree of crystallinity. The resolution of the confocal microscope under imaging conditions is on the order of 1 µm. All fluorescence signals collected during recovery monitoring thus arise within or very near the cellulose sheet.

By using hydropulping mechanism, it is possible to obtain an adequate separation of cellulose and polyaluminum. According to the results, CLSM technique is recommendable for to observe the surface morphology of a recycling product.

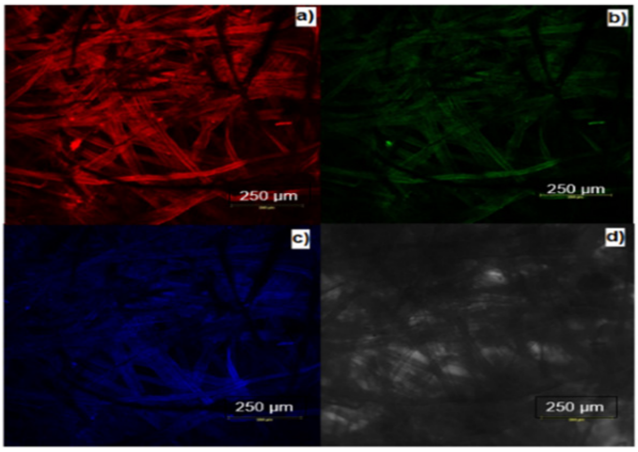

Fig. 7. CLSM images of recovered cellulose of Tetra Pak packaging.

3.3 Polyaluminum

3.3.1 Scanning Electron Microscopy (SEM)

Morphology of Polyaluminum film obtained from Tetra Pak® is shown in Figure 8. Polyethylene and aluminum appears as a single matrix. In this, well-defined channels corresponding to aluminum material as well as different particle sizes of polyethylene adhered to the matrix are observed. Moreover, some scrapped cellulose particles are seen.

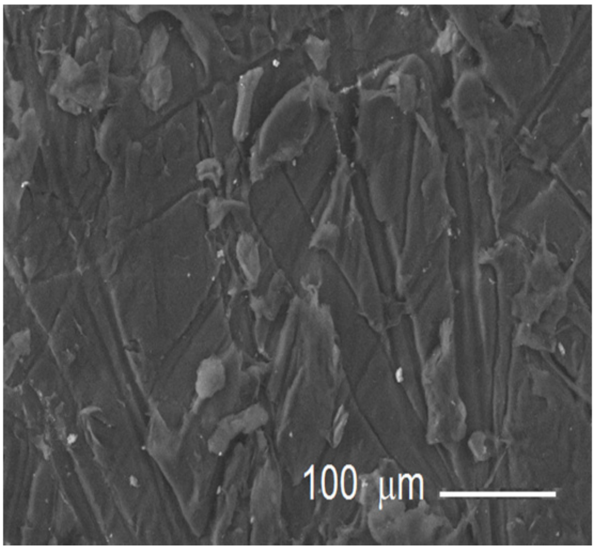

Fig. 8. SEM image of Polyaluminum film of Tetra Pak packaging.

3.3.2 Atomic Force Microscopy (AFM)

Atomic Force Microscopy images are observed in Figure 9. As can be noticed in Figure 9a non-uniformity on the relief is present. Blue color areas are below 1.35 µm, while highest peaks varying from 3.65 to 5.62 µm are observed in Figures 9b and 9d. Figure 9c corresponds to a sweep of the top of the sample, where cellulose is still present.

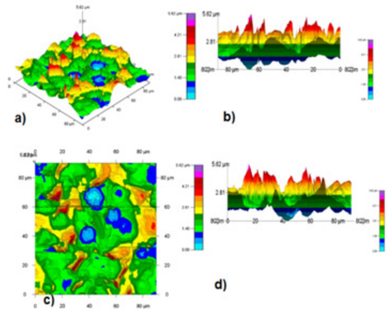

Fig. 9. AFM images of different sections of polyaluminum fromTetra Pak packaging: (a) Isometric; (b) Side; (c) Top; (d) Frontal.

3.3.3 Confocal Laser Scanning Microscopy (CLSM)

CLSM images of polyaluminum are observed in Figure 10. Brighter color corresponds to polyethylene material, while dark color to aluminum. It is difficult to decipher in detail the morphology of such combined material when using this kind of microscopy technique.

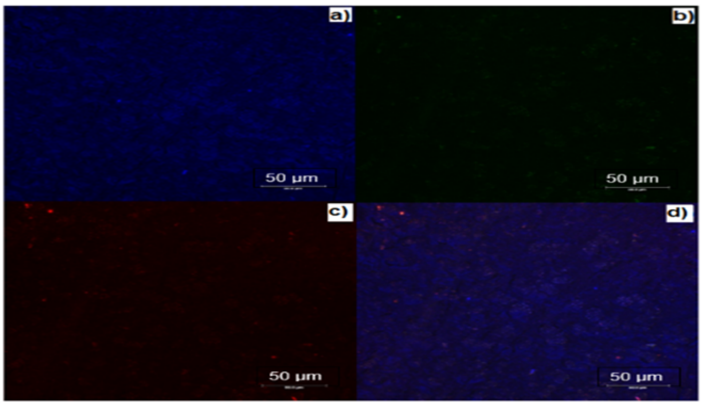

Fig.10. CLSM images of recovered polyaluminum of Tetra Pak packaging.

3.4 Aluminum

3.4.1 Scanning Electron Microscopy (SEM)

According to the SEM image in Figure 11, recovered aluminum show a soft and homogeneous surface. It is difficult to distinguish some kind of particles, cracks, voids, etc. It is to say, highly crystallinity is observed for aluminum.

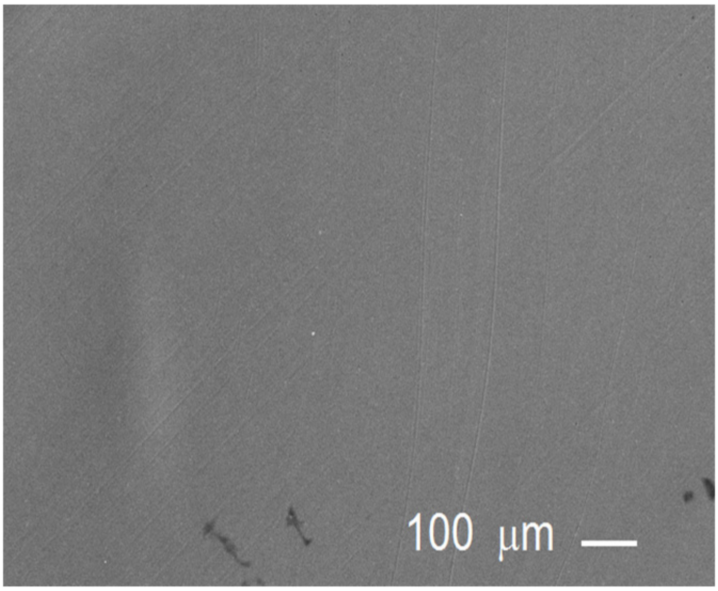

Fig. 11. SEM image of recovered aluminum obtained after incinerating of Tetra Pak® packaging.

3.4.2 Atomic Force Microscopy (AFM)

At difference of the images seen by SEM, in the AFM images of recovered aluminum (Figure 12), it is possible to observe a non-homogeneous surface, where very detailed regions are predominant. Such regions can be due to the incineration of polyethylene. The highest regions have a height of 2.48 µm (indicated in red color), while the lowest have 2 µm (blue color regions). Such differences in height indicate non uniformity of the surface. Moreover, 1.86 µm in average is found for recovered aluminum.

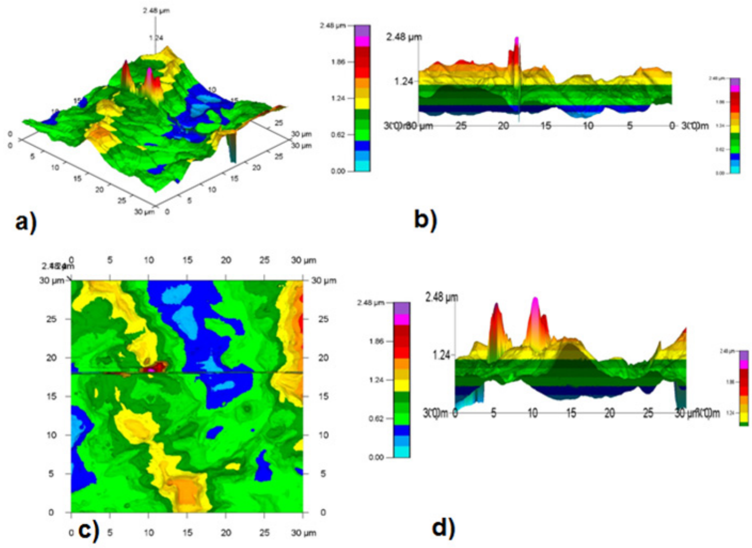

Fig. 12. AFM images of different sections of recovered aluminum from Tetra Pak packaging: (a) Isometric; (b) Side; (c) Top; (d) Frontal.

3.4.3 Confocal Laser Scanning Microscopy (CLSM)

In the case of CLSM images for recovered aluminum (Figure 13), it is possible to observe some degree of porosity, which is not visible when SEM technique is used for such purpose.

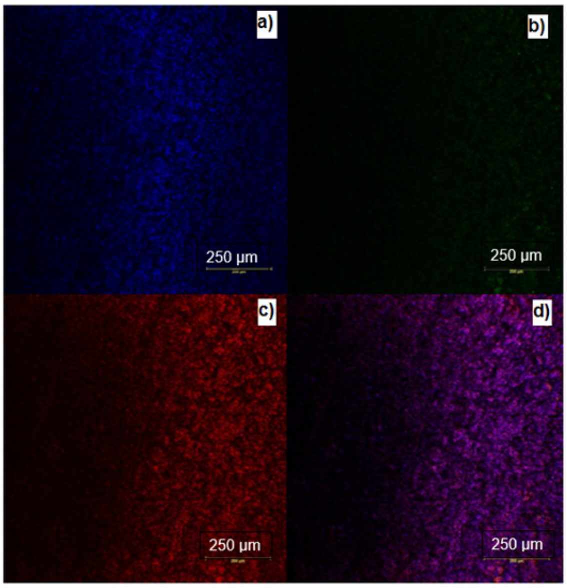

Fig. 13. CLSM images of recovered aluminum of Tetra Pak packaging.

Acknowledgements The authors acknowledge the work of MSc. Melina Tapia for the AFM analysis realized at CCIQS UAEM-UNAM. The authors recognize the support of CONACYT through the SNI program.

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