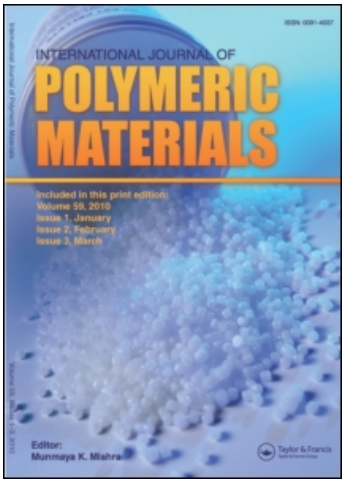

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Influence of the Preparation Technique on the Morphological Characterization of Polymeric Blends

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Influence of the Preparation Technique on the Morphological Characterization of Polymeric Blends

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Morphology-property relationships are among the parameters which determine the practical use of polymer blends. However, very few reports in the literature realize that the preparation technique of the samples for being examined by a particular methodology, is crucial for the final results. Indeed, in the case of Electron Microscopy, for instance, for the studies of polymer and copolymer blends, very little care is taken in analyzing the effect of, for example, using dissolution for preparing samples or how a microtomy process could affect the observation. Accordingly, in this work a study of the influence of different preparation techniques for SEM (Scanning Electron Microscopy) analysis of Ethylene Vynil Acetate/Polypropylene blends (EVA/PP), including a discussion on the relevance of various physical or chemical factors affecting the observation, is presented.

Keywords: Polymer blends; morphology; SEM; ethylene vinyl acetate/polypropylene

INTRODUCTION

The detailed study of the morphology of polymer blends and other characteristics such as the effect of blend composition, effect of shear stress on extrudates, the results of dynamical mechanical properties,

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etc. up to now, has shown a scarce relationship between the preparation methodology used and the results obtained.

Moreover, does not exist a systematic procedure for preparing polymer blends for microscopy work. Some authors for example, have prepared their samples by utilizing liquid nitrogen and by using the fracture technique to observe the fracture surface [1–5]. Some other authors, do fracture the samples and dissolved one of the polymer blends phases, to observe the remaining matrix [6–13]. Some others use the ultramicrotome to cut the pre-prepared sample, and then dissolve the [14–19]. A similar technique was used to keep a blend immersed in a solvent for 48 h. to extract one of the phases [20]. Other authors use the polymer blend fracture, obtained prior to the mechanical testing, and they have observed the resulting fracture surface [21, 22]. On the other hand, there exist very few reports in which the authors take into account the extrusion conditions to prepare the samples [23].

Accordingly, in this work we have studied the influence of different preparation techniques for SEM analysis, and discussed the relevance of various physical and chemical factors affecting the observation.

EXPERIMENTAL

A series of EVA/PP blend (Ethylene Vynil Acetate/PolyPropylene) in the following compositions were prepared by double extrusion, 20/80%, 40/60%, 70/30% (EVA/PP). According to a method previously reported by Maciel *et al.* [24]:

Five techniques were employed for preparing the samples for SEM observation, in two machines namely a JEOL 5200 and a Zeiss DSM940A. In the first method (*A*), the samples were pyramid-shaped cut and immersed for 48 hr in a OsO_4 solution (1%), and then ultramicrotomy cuts were obtained from the tip of the pyramid that was ready to be observed as well as from the sides.

The second technique (*B*), consisted in hand-fracturing specimens previously frozen in liquid nitrogen. The fracture was produced both perpendicularly and along the extrusion direction. The fracture surface produced was then observed in the microscope.

Method *C* consisted in forming the pyramid, freezing it with the ultracriomicrotome and obtaining ultramicrotomy cuts until a mirror-like finish is attained. The cuts were perpendicular to the extrusion direction.

Method *D* consists in dissolving the blends in toluene to extract one the two phases and to analyze by SEM the resulting structure. Finally, method *E* utilizes the fracture surface obtained prior to the mechanical testing.

RESULTS AND DISCUSSION

Technique *A* shows that the osmium tetraoxide treatment is not enough to obtain an adequate cut under laboratory conditions, since this technique produces microscopy images, as shown in Figures 1, 2 and 3, in which the contrast is very poor and no difference among phases is observed. Figures 1(a) through 1(c) correspond to specimens obtained from the upper and from the two sides respectively, of samples 20% EVA prepared by method A; likewise, the series of Figures 2 and 3 correspond to the speciments with 40% and 70% EVA content, respectively. By comparing the images, it is clear that the morphologies observed, strongly depend on where the specimens were taken from indicating the feasibility of performing anisotropy studies by Scanning Electron Microscopy (SEM).

Method *B* provides more complete information on the way the polymers were blended as well as on the morphological organization of each blend. In this second technique, it is possible to take into account the extrusion and the effect of the glass transition temperature (T_g), since the cuts are obtained at very low temperatures, as seen in Figures 4–9. Figure 4 (20% EVA, perpendicular fracture) shows the matrix of PP forming irregular leaflets with small spherical particles. Figure 7 (20% EVA), along the fracture badges formed by arranged stripes. Figure 5 (40% EVA, perpendicular the fracture), the morphology is very different, showing a matrix with little steps. Figure 6 (70% EVA), along the fracture the number of observed particles is not the same as in Figure 6.

When the fractures are compared to each other, they show a clear difference when the fracture is either along the extrusion direction or

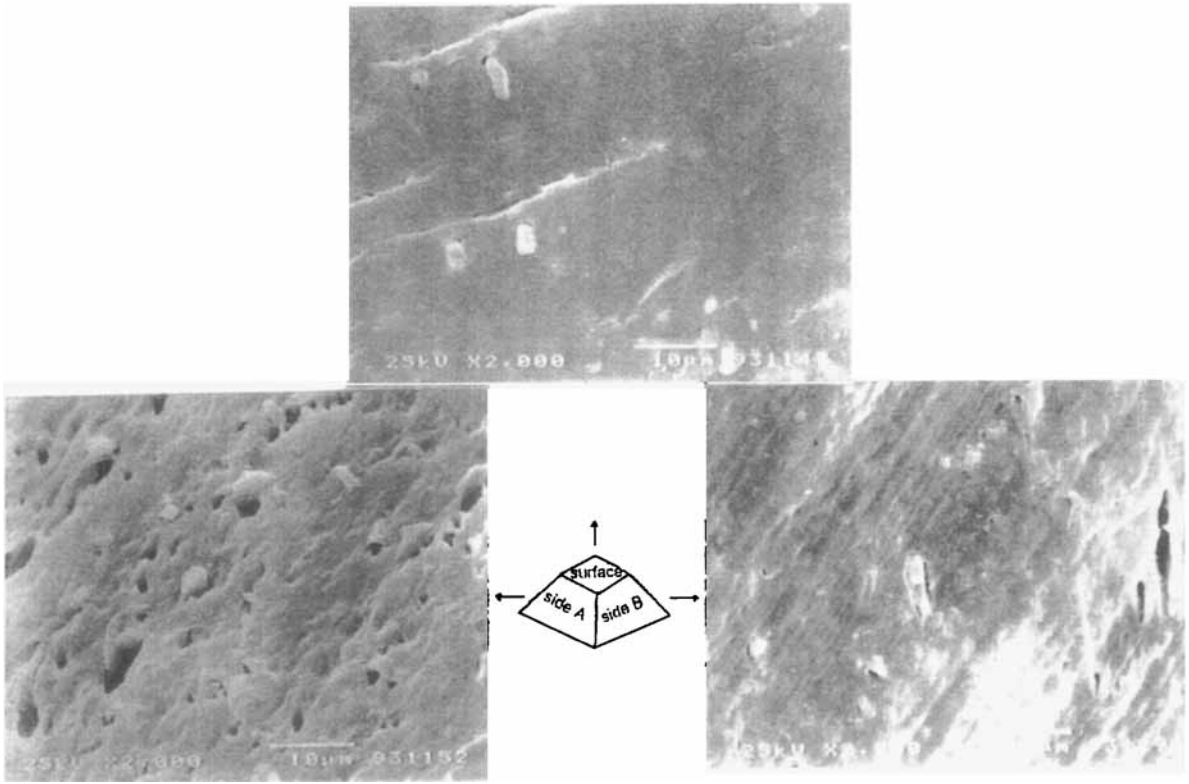

FIGURE 1 Method *A*(pyramid-shaped) 20% EVA 1s pyramid surface micrograph 1a pyramid side *A*1b pyramid side B.

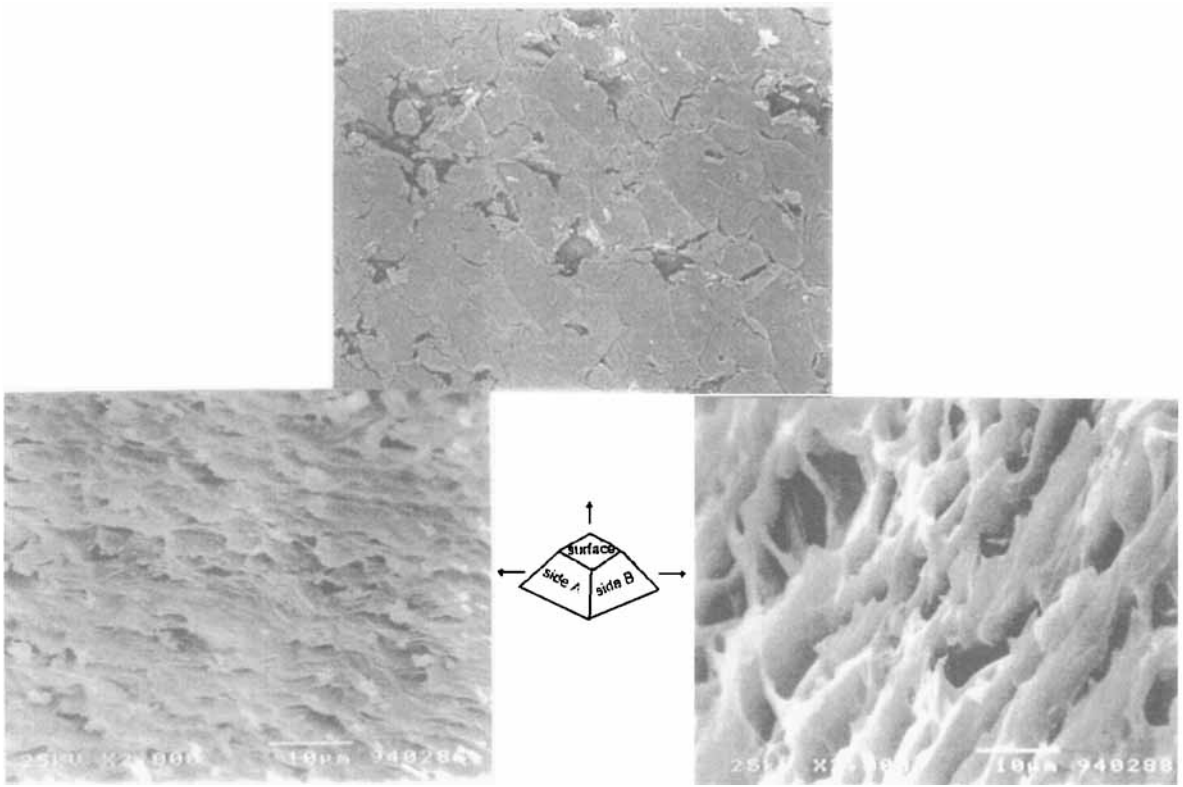

FIGURE 2 Method *A*(pyramid-shaped) 40% EVA 2s pyramid surface micrograph 2a pyramid side *A*2b pyramid side *B*.

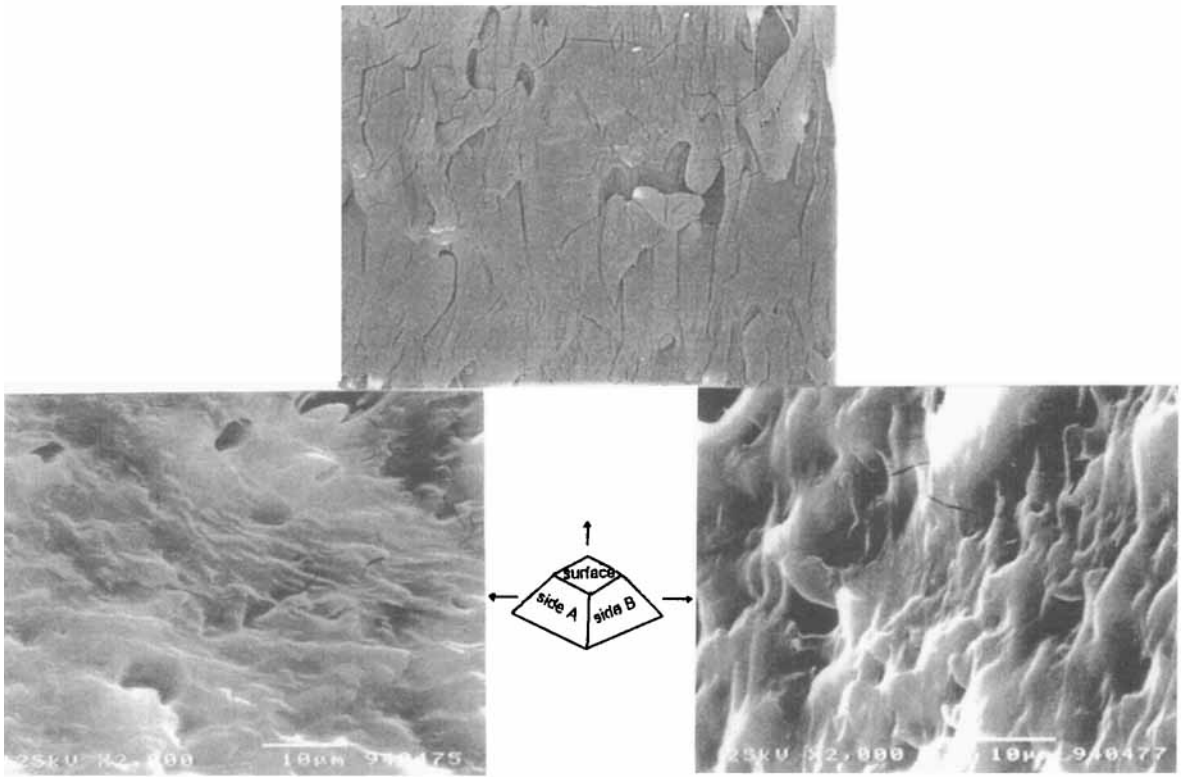

FIGURE 3 Method *A*(pyramid-shaped) 70% EVA 3s pyramid surface micrograph 3a pyramid side *A*3b pyramid side *B*.

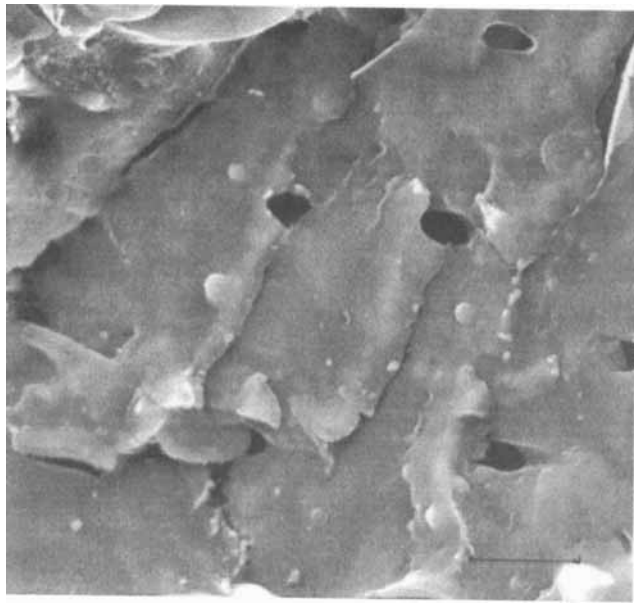

FIGURE 4 Method *B*(hand-fracturing) 20% EVA perpendicular extrusion direction.

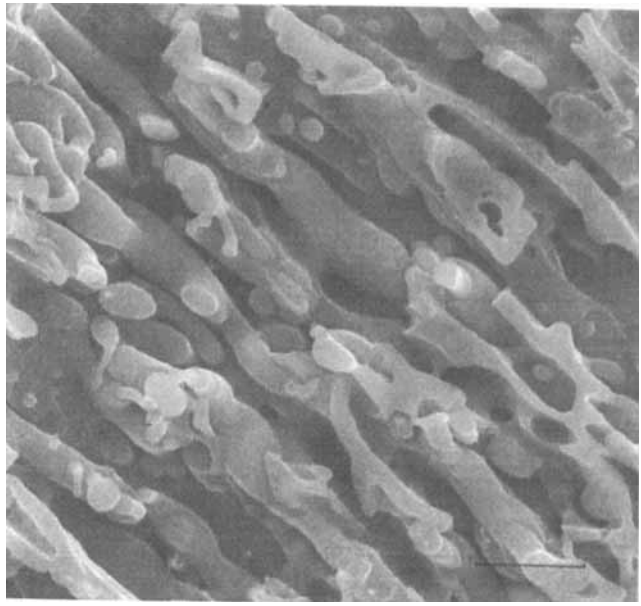

FIGURE 5 Method *B*(hand-fracturing) 40% EVA perpendicular extrusion direction.

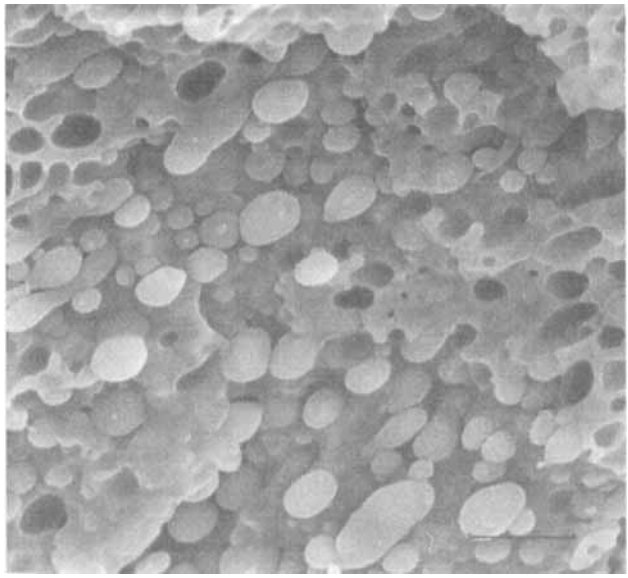

FIGURE 6 Method *B*(hand-fracturing) 70% EVA perpendicular extrusion direction.

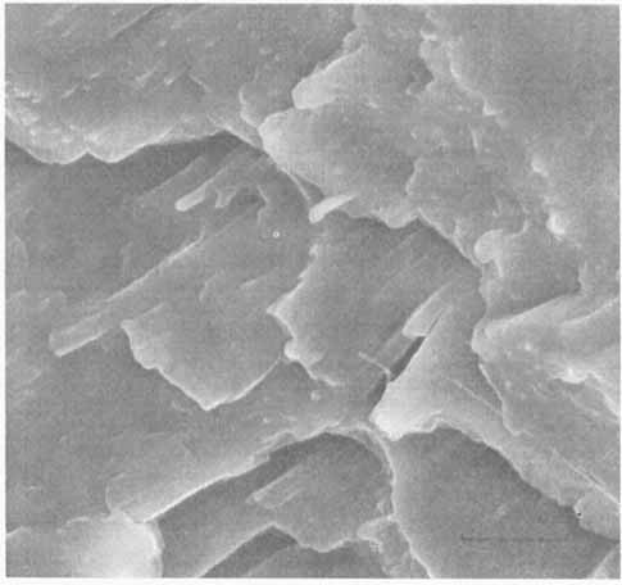

FIGURE 7 Method *B*(hand-fracturing) 20% EVA along the extrusion direction.

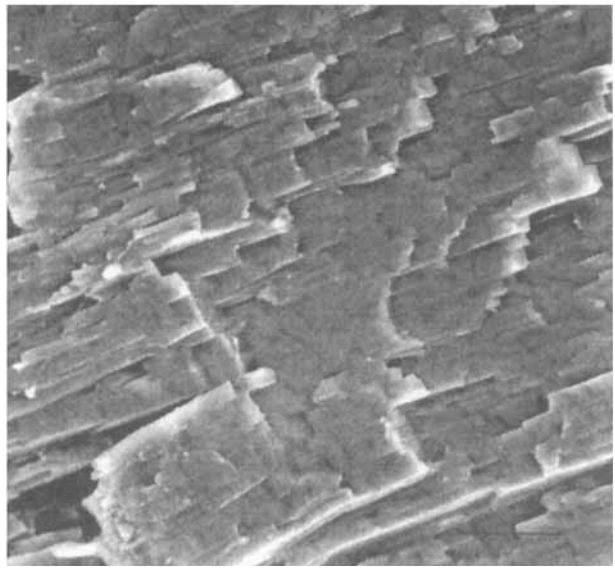

FIGURE 8 Method *B*(hand-fracturing) 40% EVA along the extrusion direction.

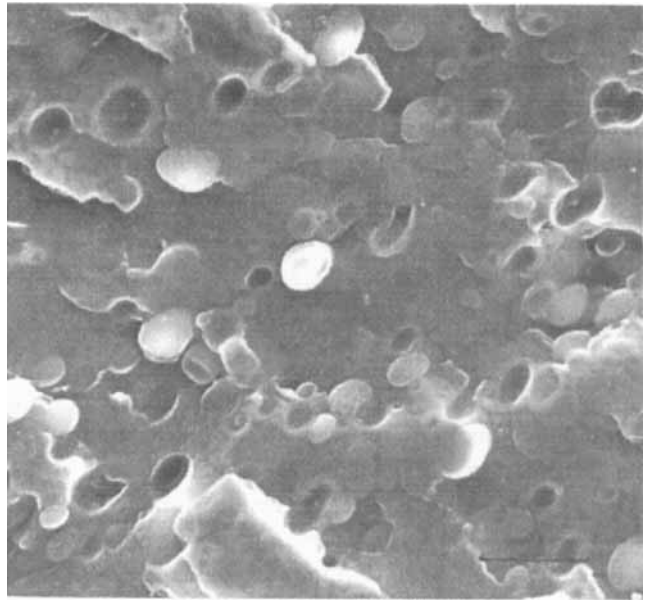

FIGURE 9 Method *B*(hand-fracturing) 70% EVA along the extrusion direction.

perpendicular to it. For example, we observe in Figure 7 that the structure consists in cylindrical forms, whereas in the Figure 8 this cylindrical shapes are not observed because in this case the fracture was against the extrusion direction.

Technique *C* (ultramicrotomy) is illustrated in Figure 10 (20% EVA), showing a dark matrix with rounded features. In Figure 12 (70% EVA), the matrix is clear and the circles are dark. In Figure 10 the matrix observed is formed by PP and the clear zones correspond to EVA. The opposite is shown in Figure 12. In Figure 11 (40% EVA) it is difficult to distinguish between both phases and clear lines along the matrix are observed.

Technique *D* (dissolution) is illustrated in Figure 13 (20% EVA) which shows the matrix with different crevice sizes, as compared to Figure 4 (20% EVA without dissolution) where a disordered matrix is observed. Figure 14 (40% EVA) and Figure 5 (40% EVA without dissolution), show a strong deformation. Figure 15–18 (70% EVA) shows a deformed structure with little useful contrast.

By the *D* technique some circular spots that correspond to one of the phases in the blends were observed, although the information is very limited because it is restricted only to one phase and the materials could have been damaged by the solvent. Also, the information is limited to one surface and, if one compares this to the fracture techniques, more information about the bulk structure and the spatial distribution of the blends, can be attained by the latter methodology.

CONCLUSION

The results of this work show the clear differences obtained in the morphology observations when different techniques are employed. The information obtained by each methodology is quite different, making complicated to compare results obtained by different authors who have used other techniques. The above indicates the convenience of reaching some sort of international agreement or standard for morphology analysis in polymeric materials.

Finally, we have observed that hand-fracturing is a very random process since the stress applied is practically out of control. This

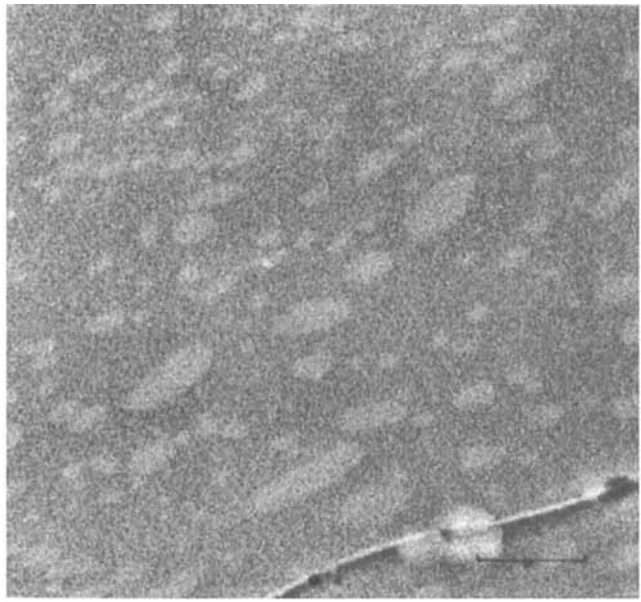

FIGURE 10 Method *C*(ultracriomicrotomy) 20% EVA cut perpendicular to the extrusion direction.

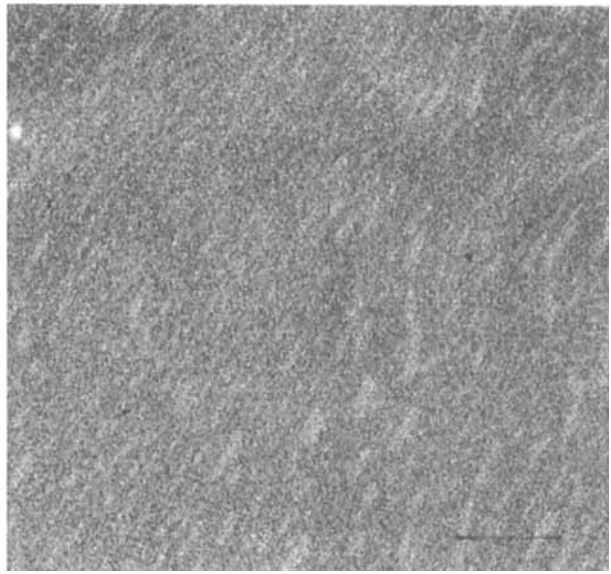

FIGURE 11 Method *C*(ultracriomicrotomy) 40% EVA cut perpendicular to the extrusion direction.

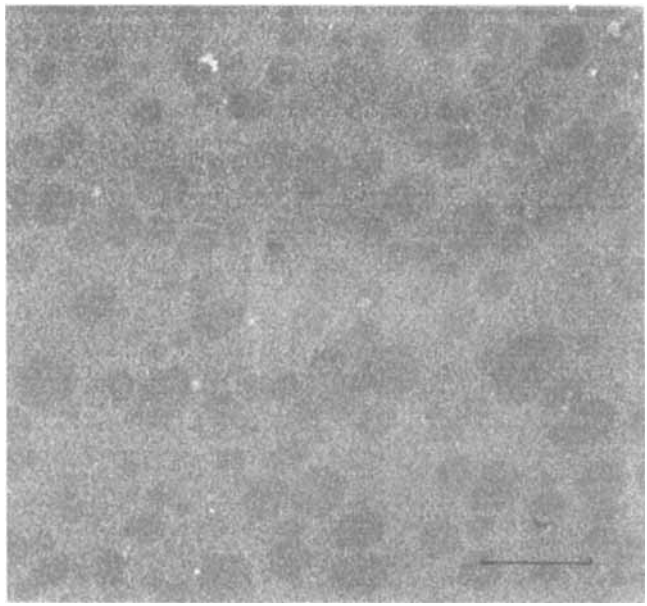

FIGURE 12 Method *C*(ultracriomicrotomy) 70% EVA cut perpendicular to extrusion.

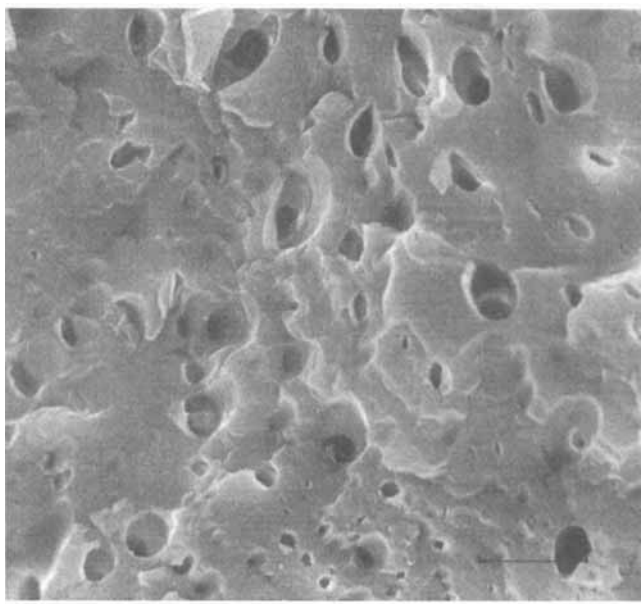

FIGURE 13 Method *D*(dissolution) 20% EVA.

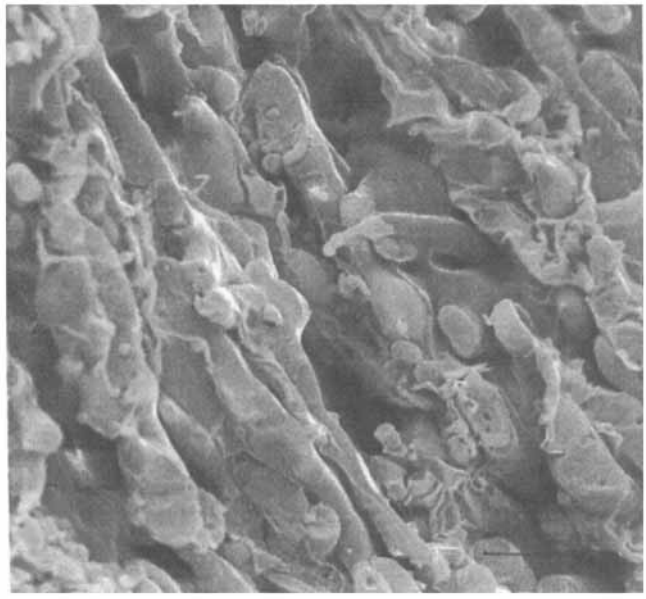

FIGURE 14 Method *D*(dissolution) 40% EVA.

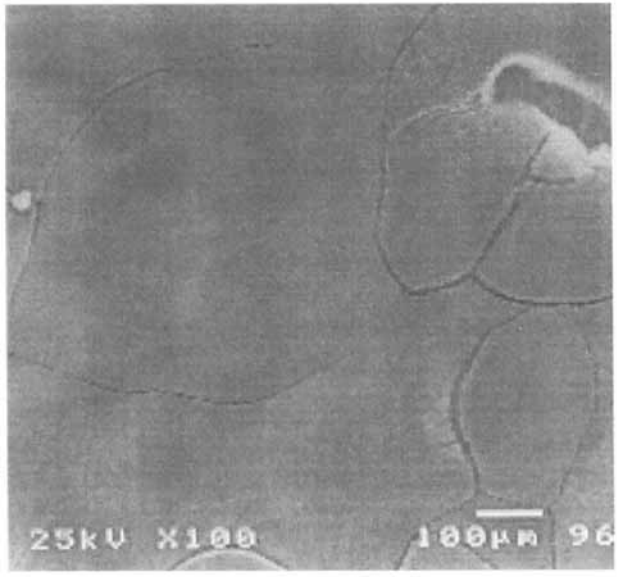

FIGURE 15 Method *D*(dissolution) 70% EVA.

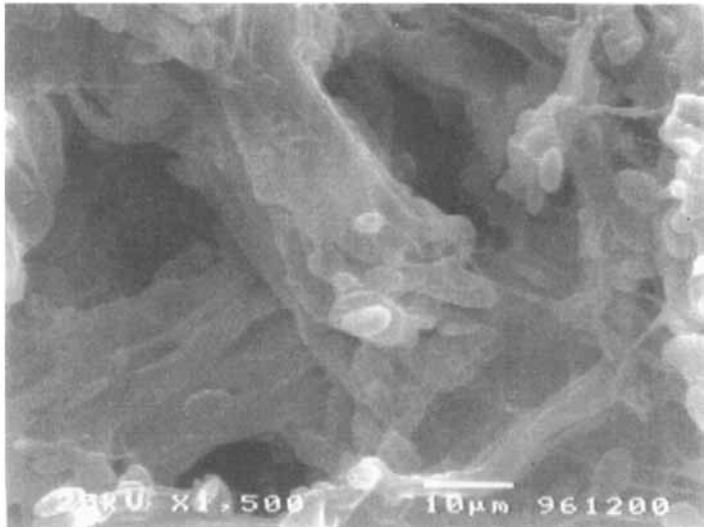

FIGURE 16 Method *E*(mechanical testing) 20% EVA.

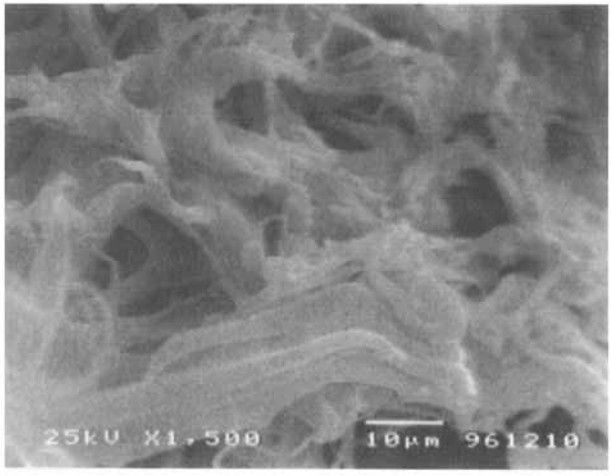

FIGURE 17 Method *E*(mechanical testing) 40% EVA.

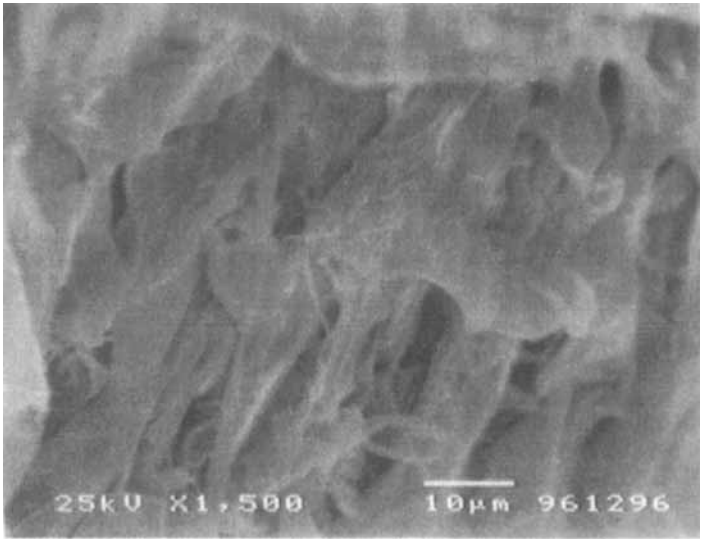

FIGURE 18 Method *E*(mechanical testing) 70% EVA.

implies that the use of automated devices must be a standard practice in sample preparation procedures.

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