

# Microwave Plasma-Initiated Grafting of Acrylic Acid on Celgard 2500 Membrane to Prepare Alkaline Battery Separators—Characteristics of Process and Product

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Received 26 June 2009; accepted 4 October 2009

DOI 10.1002/app.31596

Published online 10 December 2009 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** A process of plasma-initiated grafting of acrylic acid on commercial porous polypropylene membrane was studied. The influence of parameters of the plasma (power, gas pressure, time plasma-sample distance, sample arrangement) and grafting (solvent composition, monomer concentration, time, inhibitor presence) on the degree of grafting, amount of homopolymer produced and surface electrical resistance was determined. A degree of grafting up to 18 mmol/g was obtained, which resulted in sample resistance as low as 30 m $\Omega$  cm<sup>2</sup>. The molecular weight of AAc homopolymer that can be assumed as

equal to the MW of grafted chains, ranged from 25,000 to 50,000,000 da. SEM and water permeability measurements show that grafting causes filling of the pores, which, however, does not stop K<sup>+</sup> ions from penetrating the membrane. The performance of nickel-cadmium cells with acrylic acid grafted membranes as separator is also presented. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 868–875, 2010

**Key words:** porous polypropylene; UV polymerization; grafting degree; surface resistance

## INTRODUCTION

Surface grafting offers polymers with a new, stable, and functional surface. The literature dealing with this subject is enormously large—every year hundreds of articles appear describing grafting of various monomers on solid surfaces. To a large extent, the works described are directed to alteration of ordinary polymers. Polypropylene (PP) as a popular hydrophobic and inert polymer is often the object of hydrophilization and functionalization via grafting. Among many grafting monomers, the most popular is undoubtedly acrylic acid (AAc),<sup>1–14</sup> lately used also in grafting of binary monomer mixtures<sup>15,16</sup> or in two-step grafting with other monomer.<sup>17–21</sup> AAc is known to be a cheap and easily polymerizable monomer that introduces onto the modified surface many carboxyl polar functionalities that are pH sensitive and capable of further chemical reactions creating new functional groups.

Grafting is most often initiated with the help of high-energy radiation<sup>2,8,12,15–18,20,21</sup> but also with heavy ion beams,<sup>6</sup> UV radiation,<sup>3,10,13</sup> plasma,<sup>4,5,9,11</sup>

or a radical initiator.<sup>7,14</sup> Products of AAc-grafted PP were applied to produce cation-exchange<sup>5,15</sup> or “intelligent”<sup>17,18,20,21</sup> materials to improve the adhesion,<sup>2</sup> to decrease the deposition of bacteria<sup>16</sup> or protein,<sup>13</sup> to enhance the gas transport through PP film,<sup>3</sup> to introduce functional groups for better dyeability,<sup>14</sup> or for immobilization of biomolecules.<sup>1,11</sup> One can also find a few articles that authors tried to use grafted nonwoven fabrics of polyolefines (PP,<sup>8</sup> polyethylene (PE),<sup>22</sup> and PE-PP<sup>23</sup>) as battery separators.

Our previous study<sup>24–26</sup> on the use of PP porous membrane grafted with AAc showed that it can serve as an effective separator for high-power alkaline batteries and replace the commonly used, but much less chemically resistant, cellulose, which shows gradual loss of tensile strength and decrease of molecular mass. The goal of this article is to find out how the process parameters affect the properties of PP membrane and to get the surface resistance of the membrane lower than 100 m $\Omega$ /cm<sup>2</sup>, making it suitable as a battery separator. Through the additional experiments testing the high-performance batteries [(high-rate chargeability test (HRC) and high-rate dischargeability test (HRD)], we wanted to check the ability of nickel-cadmium cells equipped with the new separator to accept charge and allow a high rate of discharge. In these tests, the HRC was evaluated by discharging the cell at low current, after charging

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it at various increasingly high currents, and HRD was evaluated by charging the cell at low current and then discharged at different increasingly high current densities; the higher the HRC and HRD are, the better is the cell performance.

## EXPERIMENTAL

### Materials and equipment

Microporous PP membrane—Celgard 2500 (Daicel Chemical Industries)—was used as a substrate for grafting. Its thickness was 25.4  $\mu\text{m}$ , pore size was  $0.05 \times 0.20 \mu\text{m}$  and porosity of 45%. The membrane was cut into  $5 \times 5 \text{ cm}^2$  pieces, cleaned by rinsing in methanol, and then dried between two layers of filter paper in air at room temperature. Acrylic acid (AAc) was purchased from Fluka and vacuum-distilled before use. All other chemicals were supplied by POCh, Poland. Mili-Q water was used in all experiments.

A microwave plasma generator of 2.45 GHz frequency with power regulated from 0 to 1000 W (Ertec, Wroclaw, Poland) was used throughout the study. Remote plasma was generated in a quartz tube at the top of a thick-walled glass chamber.

### Procedures of plasma treatment and sample grafting

The PP sample was attached to the reactor table set in the postdischarge area at a set distance from the plasma edge. The reactor was then evacuated to a pressure below  $10^{-3}$  mbar (0.1 Pa), and the argon flow was adjusted to give a pressure of 0.5 mbar (50 Pa). The plasma was ignited at a selected power and kept for the time required. After plasma treatment, the chamber was filled with air and the membrane was allowed to stay in air for 10 min. After that, it was immersed in AAc solution (earlier flushed with argon for 10 min) and exposed to UV radiation for a selected time. The modified sample was washed with water to remove homopolymer and dried in air. The amount of PAAc grafted onto PP and the resulting amount of homopolymer were determined gravimetrically. The degree of grafting was calculated in moles of grafted AAc per unit mass of substrate. The data obtained were taken as the average from 2 to 5 independent experiments.

### Modified sample characterization

#### Scanning electron microscopy (SEM)

The surface and cross-section of samples were observed using a JSM-5800 LV, Jeol device working at 25 kV.

### Water permeability

Membranes were immersed in ethyl alcohol (half an hour), then in ethyl alcohol/water mixture (50/50 vol %) (1 hour) and then left in water for the next day. The volume flux was determined through timed collection of permeate at 0.05 MPa.

### Measurement of surface resistance

The surface resistance ( $R$ ) of membranes was determined by two successive measurements of potential difference between the reference electrodes—without membrane ( $E$ ) and with membrane ( $E'$ ). Concentrated KOH solution (29 wt %,  $d = 1.28 \text{ g/cm}^3$ ) was used as electrolyte. Surface resistance was calculated from the equation<sup>27</sup>:

$$R = (E' - E)S/I$$

where  $S$  is the membrane surface area exposed to the electric field.

### Application of modified membranes as separators in a nickel-cadmium cell

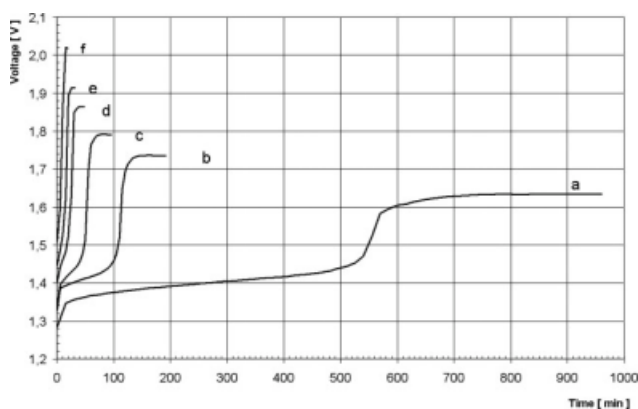
#### Nickel-cadmium tested cell

A home-made model rectangular single vented cell ( $C = 0.4 \text{ Ah}$ , one cathode and two anodes—sintered plates, cell volume =  $0.0054 \text{ dm}^3$ ) was used to evaluate separators. The positive capacity-limited cell with a capacity of 0.4 Ah was constructed using a  $4.0 \times 4.0 \text{ cm}^2$  nickel sheet electrode wrapped in the tested PP separator. It was placed between two sheets of a  $5.0 \times 5.0 \text{ cm}^2$  cadmium electrode with a larger capacity—about two times larger than that of the nickel electrode. These electrodes were then sandwiched between two thin-meshed resin plates, and the assembly was tightly bolted at the four corners. The cell was soaked and tested in an aqueous KOH solution ( $d = 1.28 \text{ g cm}^{-3}$ ).

#### Nickel-cadmium cell performance tests

The charge/discharge characteristics of the cell examined, at different charge and discharge rates, were determined at ambient temperature using a charge-discharge unit (Atlas-Sollich, Poland) connected to a PC system loaded with charge-discharge controlling software.

In the first step of investigations, the cells examined were activated by charging at the 0.1 C rate for 16 h and discharging at 0.2 C using a cut-off voltage of 1.0 V, charging at a constant current of 40 mA (0.1 C), discharging at constant current of 80 mA (0.2 C). The cells were then charged at the 0.2 C rates for 8 h and discharged at 0.2 C and 1.0 V. This process was repeated for four times. After this, the tested cells were charged by the constant current charging



**Figure 1** An example of charge curves for tested nickel-cadmium cells, charged at: (a) 0.1 C, (b) 0.5 C, (c) 1.0 C, (d) 2.0 C, (e) 3.0 C, and (f) 5.0 C; electrolytic resistance of separators used:  $42 \text{ m}\Omega \text{ cm}^2$ .

method at 0.1 C, 0.5 C, 1 C, 2 C, 3 C, or 5 C to obtain a fully charged state (160 %), (see Fig. 1) and then discharged at 0.2 C to 1.0 V, where C is the numerical value of capacity of the tested cell [Ah]—HRC. It was calculated as:  $\text{HRC}(\%) = (C_i/C_{80}) \times 100$ , where  $C_i$  and  $C_{80}$  are the discharge capacity after charging at “i” and 80 mA, respectively. In the second step, the cells were charged by the constant current (80 mA) charging method at 0.2 C to obtain a fully charged state (160 %) and then discharged by the constant current discharging method at 0.1 C, 0.2 C, 0.5 C, 1.0 C, 2.0 C, 3.0 C, and 5.0 C using a cut-off voltage of 1.0 V—(HRD).

HRD was evaluated as:  $\text{HRD}(\%) = C_i/(C_i + C_{80}) \times 100$  where  $C_i$  is the discharge capacity at a current  $i$  and  $C_{80}$  the discharge capacity when the cell is discharged at 80 mA just after discharging at “i” mA.

### Homopolymer viscosity measurement

The grafting solution after removing the membrane was evaporated at room temperature followed by vacuum drying. The homopolymer obtained was weighed, and its amount was calculated as a mass fraction of monomer used. The viscosity of the homopolymer was determined in 2M NaOH solution at 25°C. Automatic viscometer (VB2 Lauda) was used. The intrinsic viscosity was estimated by one point measurement of the viscosity at a concentration of  $0.04 \text{ g}/100 \text{ cm}^3$ , using the Salomon-Ciuta equation.<sup>28</sup> Molecular weight was calculated taking  $K = 1.5 \times 10^{-3}$  and  $\alpha = 0.54$ .<sup>29</sup>

## RESULTS AND DISCUSSION

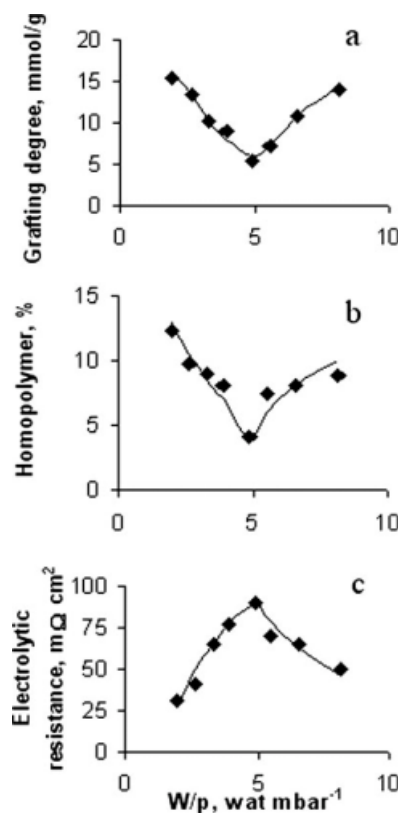
### Dependence of grafting results on plasma parameters

The plasma initiated process of grafting is dependent to a great extent on the plasma device and

plasma parameters. In the case of this study, a microwave plasma generator was used, and the relationship between the results (grafting yield and area resistance) and plasma parameters—plasma power, gas pressure, treatment time, sample-to-plasma edge distance—was determined.

According to Yasuda,<sup>30</sup> for nonpolymerizable gas plasma significance is given to the composite parameter  $W/p$ , where  $W$  is the plasma power and  $p$  is the gas pressure. The sensitivity of grafting effects to this composite parameter is shown in Figure 2. In the selected range of the  $W/p$  parameter, the degree of grafting [Fig. 2(a)] and amount of homopolymer obtained [Fig. 2(b)] show the minimal value as  $\sim 5 \text{ W/Pa}$ . In such conditions, the amount of radical created must be small, which results in a small amount of grafted acrylic acid, hence high surface resistance [Fig. 2(c)].

From the processing point of view, the next important parameter of plasma treatment is the activation time. For the  $W/p$  parameter equal to  $5.5 \text{ W/Pa}$ , prolongation of plasma action from 0.5 to 1 min results in significant increase of grafting degree (from 4 to 11 mmol/g), but later on this dependence is very weak; and for 4 min of plasma treatment, grafting degree reaches  $\sim 12 \text{ mmol/g}$ . The amount of homopolymerized acrylic acid increases from 5 to



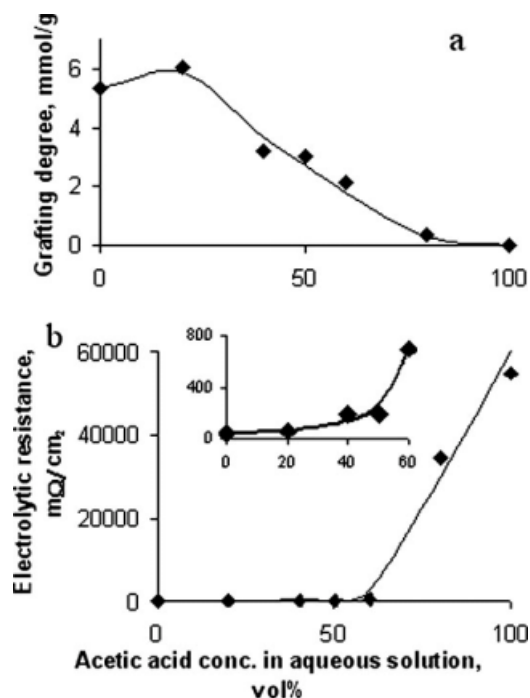
**Figure 2** Effect of composite plasma parameter (power/pressure) on grafting degree (a), amount of homopolymer (b), and surface resistance (c).

**TABLE I**  
Dependence of Grafting Results on the Sample Arrangement. Plasma-Sample Distance: 80 mm

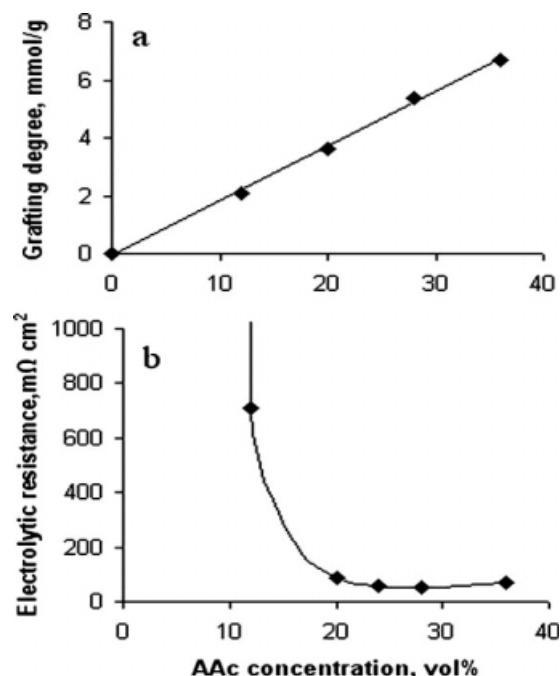
Sample arrangement	Grafting degree (mmol/g)	Electrical area resistance ( $m\Omega\text{ cm}^2$ )
Two-side treatment, sample lies on the table	9.6	80
Two-side treatment sample hanging (suspended)	10.3	53
One-side treatment sample lies on the table	9.3	547
One-side treatment sample hanging	9.8	46

12%. In such circumstances, the surface resistance, which is depended to grafting yield mostly, drops down rapidly till 1 min of activation and then slightly decreases with the time of the process. Taking the above into consideration, it seems that 1 min is the sufficient time for membrane activation.

In the case of remote plasma, there is one more process parameter whose effect should be evaluated. It is the distance between the plasma edge and the modified sample. As one might expect, when the sample is far away from the plasma, the efficiency of grafting and the amount of polymerized poly (acrylic acid) become smaller. Both these trends are comprised in the surface resistance.



**Figure 3** Effect of acetic acid concentration on grafting degree (a) and surface resistance (b). Other grafting parameters: grafting time, 5'30"; AAc concentration, 20 vol %.



**Figure 4** Dependence of grafting degree (a) and electrolytic resistance (b) on acrylic acid concentration in grafting solution. The other grafting parameters: acetic acid/water = 1 : 1, grafting time, 5'30".

In all the aforementioned experiments, the samples were treated on both sides. However, taking into account the membrane thickness, we decided to check if one-side modification is enough for preparation of acceptable battery separators. This would be an advantageous simplification of the activation process. Two ways of sample localization in the chamber were evaluated: in the first one, the sample was put on the table and in the second one, the sample was freely hanging in the reactor. In both cases, the sample was in the same distance from the plasma edge. The results of these studies are shown in Table I. It is clearly seen that a high amount of grafted PAAc might be obtained from any arrangement. However, to be sure, to obtain the required electrical properties, the sample has to be treated on both sides or plasma gas can freely flow through the sample.

**TABLE II**  
Effect of Grafting Time on Grafting Degree and Surface Properties of PP Membrane

Grafting time (min)	Grafting degree (mmol/g)	Surface resistance ( $m\Omega\text{ cm}^2$ )
0	0	130,000
2.5	0.74	18,000
4	7.42	80
5.5	9.16	51

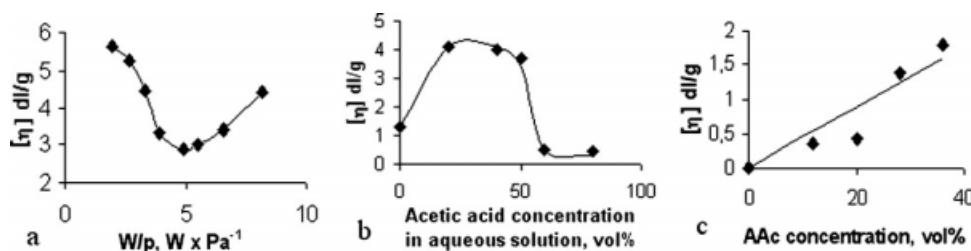


Figure 5 Dependence of intrinsic viscosity of homopolymer on some process parameters.

### Dependence of grafting results on grafting parameters

Grafting parameters and the previously mentioned plasma parameters can affect the final result of the modification process. In this step, we kept the plasma parameters constant while changing the conditions of grafting—solvent composition, acrylic acid concentration, grafting time, and presence of homopolymerization inhibitor.

In the AAc grafting process, an aqueous solution of acetic acid was taken as the solvent. In the opinion of some investigators<sup>6,21,23</sup> the lowering of the pH value of the grafting solution has a positive effect on the acrylic acid grafting. The process of acrylic acid grafting goes well in water alone. However, the addition of a small amount (16 vol %) of acetic acid seems to increase the degree of grafting [Fig. 3(a)]. As a consequence, an improvement in surface conductivity is observed [Fig. 3(b)]. However, the progressive increase of acetic acid concentration has a negative effect on the grafting yield and on the surface resistance. For this reason, the best grafting condition to conduct the modification is 15–20% aqueous solution of acetic acid.

Looking at Figure 4(a) there is no doubt that increase of the amount of monomer in the grafting solution results in a higher degree of grafting, and this relationship is linear in the range of conditions investigated. The further increasing of acrylic acid concentration in solution, however, results in wrinkling of the polymer membrane. Taking into account the surface resistance [Fig. 4(b)], it seems reasonable to use a 20% solution of acrylic acid. A similar situation is observed with the grafting time—the longer the grafting time, the higher is the grafting efficiency (see Table II).

### Homopolymer concentration and viscosity measurements

After the process, a certain amount of acrylic acid homopolymer (up to 13% of monomer used) was found in the grafting solution. To lower the amount of homopolymer in the grafting solution seemed to be desirable. The radical inhibitors most often used in such processes are ferrous

salts.<sup>4,6,9,15,21,23</sup> In the conducted studies Mohr's salt—a stable  $Fe^{2+}$  compound—was applied. As expected, the amount of homopolymer obtained during the grafting process decreases with the increase of Mohr's salt concentration. The amount of chosen inhibitor that practically stops homopolymerization lays between 0.2 and 0.3%. The reduction of homopolymer concentration was connected, however, with lowering the degree of grafting (from  $\sim 5$  mmol/g without inhibitor to below 1 mmol/g with 0.29% of it in the grafting solution) and increasing the surface resistance of the modified membrane (Fig. 8) well above the assumed limit of  $100 \text{ m}\Omega \text{ cm}^2$ .

At the first approach, one can assume that the molecular weight of homopolymer should be comparable to the molecular weight of grafted polymer because all chains grow in the same conditions. For this reason, several samples of AAc homopolymer were evaluated for their molecular weight. Depending on the process parameters, the measured intrinsic viscosity values varied from 0.35 to 6.24. These correspond to molecular weight from 25,000 to 5,000,000 g/mol. The particular relationships of intrinsic viscosity with selected parameters are shown in Figure 5.

The presented data are in good relation to those previously found for degree of grafting. The worst conditions for grafting is to use the composite parameter of around 5 W/Pa for which the polymer chains obtained are very short. Polymerization of acrylic acid in acetic acid solution offers the longest PAAc chains for 20–40% concentration, and the molecular weight of poly(acrylic acid) became larger when more monomer was present in the grafting solution. The earlier results allow us to consider that the

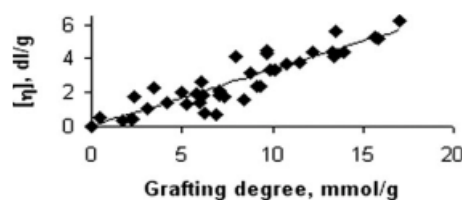
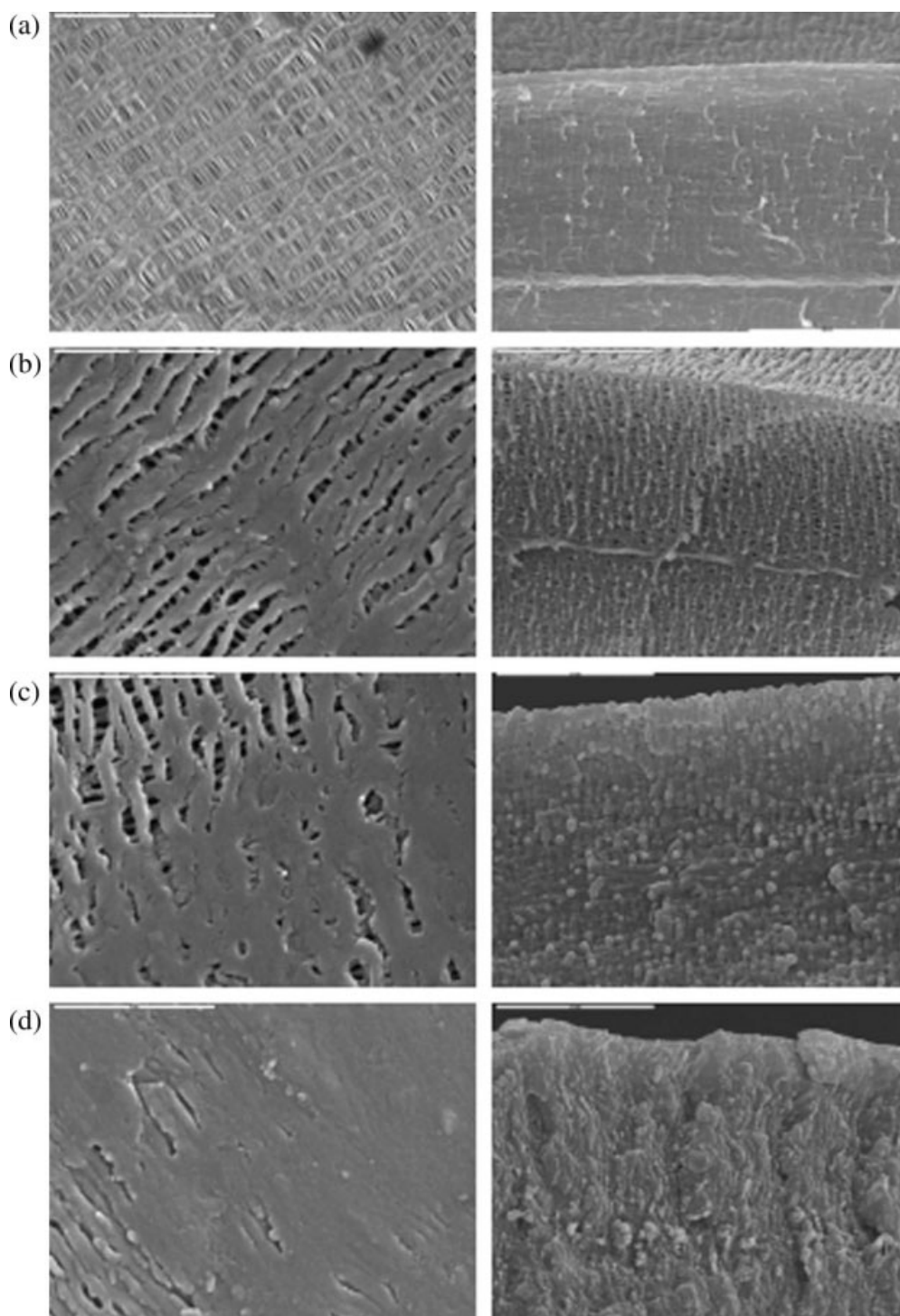


Figure 6 Correlation between the grafting degree and intrinsic viscosity of homopolymer.

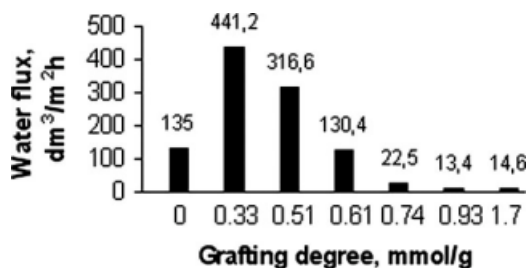


**Figure 7** SEM pictures of surface (left column) and cross-section (right column) of unmodified membrane (a) and grafted polypropylene membrane of grafting degree 2.3 (b), 9.7 (c), and 16.2 mmol/g (d). Magnification = 10,000 for surface and 5000 for cross-section.

molecular weight of grafted chains is directly related to the amount of grafted AAc. This correlation (coefficient  $R^2 = 0.83$ ) can be seen in Figure 6. Such dependence, though apparently inconsistent with the chain polymerization mechanism, was observed also by other researchers dealing with UV grafting.<sup>31</sup>

### Scanning electron microscopy

SEM pictures of neat and grafted porous PP, both surface and cross-section, are shown in Figure 7. With the increasing degree of grafting, the walls of the pores become thicker, and grafting polymer seems to cover more and more of the sample area.



**Figure 8** Water flux through the polypropylene membrane as a function of PAAc grafting degree.

Grafting takes place not only on the surface but also inside the pores, which can be seen on the micrographs of cross-section cuts.

### Water permeability

When poly(acrylic acid) is grafted onto Celgard 2500 membrane, it hydrophilizes it. However, as is seen in the SEM pictures, some pores are plugged by PAAc molecules. Hence, there is an ambiguous effect of surface grafting. To check it, the membranes obtained were evaluated for water permeability. At the beginning, when the degree of grafting is rather low, the membranes become more permeable (see Fig. 8). For larger degrees of grafting, when the PAAc chains are longer, the modified membranes become less permeable. Finally, they appear almost impermeable to water for degrees of grafting higher than 2 mmol/g. Surface resistance of values below 50 mΩ cm<sup>2</sup> is characteristic of samples with a degree of grafting higher than 5 mmol/g (see Fig. 2). This means that the best alkaline spacers should have a nonporous structure and that ion transport is conducted through pores filled with poly(acrylic acid).

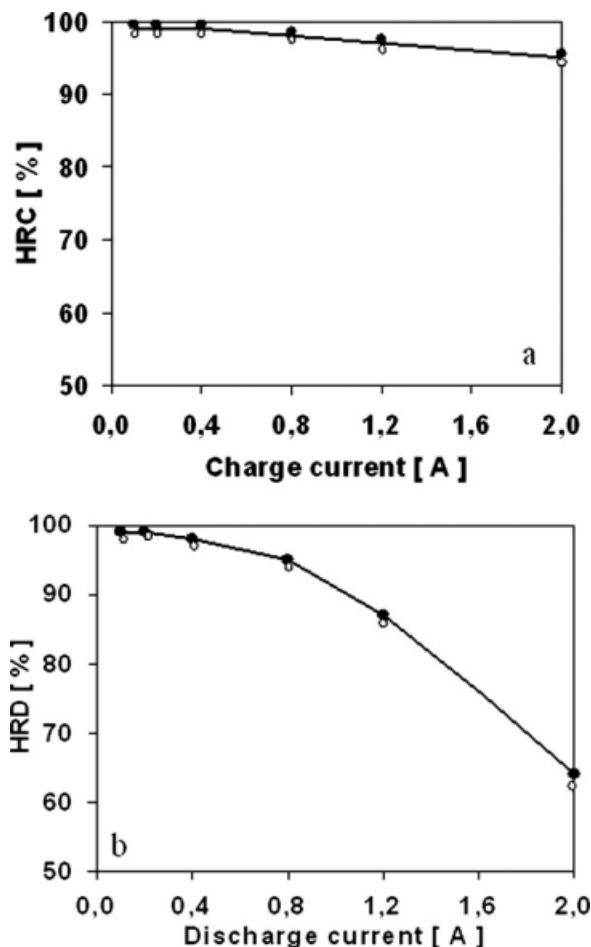
### Performance of nickel-cadmium cells with acrylic acid grafted membranes as separator

HRC and HRD were investigated for the nickel-cadmium cell with acrylic acid grafted membranes as separator at 25°C, compared to that with commercial separators (cellophane membranes). Figure 9 shows the HRC and HRD for the cells with acrylic acid grafted membranes as separator and the commercial cellophane separator (cellulose material). As can be seen from this Figure 9(a), both types of cells maintained an HRC of more than 90% even at high charge current rate such as 2.0 A (5 C). Moreover, an HRD of over 60% was retained at a high discharge current (2.0 A) even in case of the acrylic acid grafted membranes and commercial separator as shown in Figure 9(b). These results strongly indicate that the HRC and HRD of the cell with grafted membranes as separator are excellent and a little higher than those with commercial separators. The

excellent high-rate capability can be ascribed to the very low electrolytic resistance of the membranes tested, owing to the grafting of polyacrylic acid onto their surface, which makes them hydrophilic and well wet by electrolyte.

### CONCLUSIONS

All parameters used for the plasma had significant influence on the grafting process. The degree of grafting shows a minimum for  $W/p$  equal to 5 Watt/mbar. As was expected, increase of distance of the sample from the plasma edge decreases the effectiveness of grafting. The opposite effect was observed for treatment time—the longer the time, the higher was the degree of grafting obtained, but for time longer than 1 min, this increase was not significant. The dependences of amount of homopolymer on the plasma parameters were similar to those of the degree of grafting, unless the homopolymerization inhibitor was used. The presence of the latter



**Figure 9** (a) High-rate chargeability (HRC) and (b) high-rate-dischargeability (HRD) for nickel-cadmium cells with the modified polypropylene membrane (●) and commercial cellophane separator (○); electrolytic resistance of PP modified separators used: 42 mΩ cm<sup>2</sup>.

in the grafting solution significantly lowered the amount of homopolymer but also lowered the degree of grafting.

The composition of the grafting solution also plays some role in the process. The best results (from the point of view of degree of grafting) were obtained for weakly acidic aqueous solutions of high concentration of acrylic acid. This last parameter is limited, however, by the appearance of the sample for very high degrees of grafting gave wrinkled samples not usable for the purpose of the work. For that same reason, the grafting time cannot be too long, although longer time means higher degree of grafting.

Very significant is the arrangement of the sample in the plasma reactor. The best conditions are in the case when the plasma has the possibility of going through the porous sample. The radicals are created on both surfaces and inside the pores that might ensure more even grafting. Such an arrangement allows shortening of the modification time, which is important from the technological point of view.

The degree of polymerization of grafted chains is strictly connected with the degree of grafting and depends on the parameters applied. It varies from 350 to 70,000. The grafting takes place on the surface and inside the membrane pores, giving membranes impermeable to water (for degree of grafting  $>2$ ) but hydrophilic, of low area resistance.

Undoubtedly, the electrical properties of the modified membranes depend on the degree of grafting. However, with a relatively wide range of plasma and grafting parameters, one can obtain membranes of degree of grafting  $>4$  mmol/g, hence suitable for application as separators for high-power alkaline batteries.

PP membranes grafted with poly(acrylic acid) used as separators in high-power nickel-cadmium batteries ensured the excellent high-performance of the cells, which can be ascribed to their high hydrophilicity and very low surface resistance.

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