Chemical Binding of Jojoba Liquid Wax to Polyethylene

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ABSTRACT: Jojoba wax was chemically bonded to polyethylene—in film or hollow fiber form—*via* a stable sulfonamide bond. The jojoba-bonded polyethylene was obtained by binding allyl amino jojoba derivatives to chlorosulfonated polyethylene. The amount of jojoba added to the polymer ranged from 9 to 98% (w/w), depending on the reaction conditions. Swelling of the polymer in the reaction solvent was the major factor affecting the efficacy of the chemical binding of the jojoba amino groups to the chlorosulfonyl entities of the polymer. The double-bond regions in the bound jojoba wax were preserved, i.e., they were shown to be reactive in a bromination reaction. These modified membranes can find application in separation processes, such as metal ion separation and pervaporation.

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KEY WORDS: Allyl-amino jojoba derivatives, chlorosulfonated polyethylene, jojoba bound to polyethylene, jojoba wax, polyethylene.

Jojoba liquid wax (I) is a stable, highly lipophilic, nontoxic "oil" obtained from the desert plant jojoba (*Simmondsia chinensis*). This unique liquid wax differs from common vegetable oils and animal fats in that it is composed mainly (97%) of linear wax esters. More than 80% of these esters are produced from C_{20} and C_{22} monounsaturated alcohols and acids (1) (Scheme 1).

Jojoba wax, being stable to oxidation, may remain chemically unchanged for years. The main application of jojoba wax is currently in cosmetics, but the wax and its derivatives have potential commercial uses in a variety of fields, including pharmaceuticals and lubrication (2,3). It was recently found that chemical binding of jojoba wax to a polystyrene matrix (4,5) followed by sulfurchlorination or phosphonation produced a solid suitable for use as an extractant for metal ions (6). This type of extractant has a number of advantages over resins impregnated with phosphonated and sulfurized jojoba wax derivatives, which have previously been used for extraction of metal ions, such as uranium and mercury (7–9), i.e., there were no problems of loss of expensive extractants or of contamination of aqueous solutions.

The solid extractants described above were obtained in bead form, but it was thought that for ion-exchange or aqueous/organic separation applications, binding of the jojoba

$$(Z,Z)\text{-}CH_3(CH_2)_7CH=CH(CH_2)_mCOO(CH_2)_nCH=CH(CH_2)_7CH_3$$
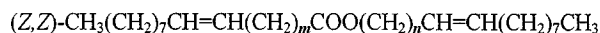
Jojoba wax (I)

m = 7, 9, 11, 13	n = 8, 10, 12, 14
average composition (%) 11, 71, 14, 1	1, 44, 45, 9

SCHEME 1

wax to polyethylene membranes or hollow fibers would be more suitable. In this paper, we thus describe the preparation of extractants in membrane or hollow fiber form by chemical binding of allyl amino jojoba derivatives to polyethylene (PE) that had previously been chlorosulfonated.

EXPERIMENTAL PROCEDURES

Materials. Crude (unbleached) jojoba wax ("Jojoba Israel," Kibbutz Hatserim, Israel) was used in all the experiments without purification [iodine number (Wijs) 84; acid number 2.7; saponification number 93]. Low-density PE (density 0.922 g/mL; thickness 50 μm; melting index 0.3) was purified before use by Soxhlet extraction with hexane. Dioxane (Frutarom, Haifa, Israel) was distilled and dried over NaOH, and dimethyl formamide (DMF, 99.8%; Aldrich, Milwaukee, WI) was dried over a 4Å molecular sieve. All other solvents were chemical pure-grade (Frutarom) and were used without further purification. *N*-Bromosuccinimide (NBS; Aldrich) was recrystallized from water before use. Hexamethylene tetraamine (hexamine) was obtained from BDH Laboratory Reagents (Darmstadt, Germany). All other chemicals (Aldrich) were reagent-grade and were used without further purification.

Elemental analysis. Elemental analysis, based on ASTM methods, was performed by the Analytical Laboratory of the Institutes for Applied Research (Ben-Gurion University of the Negev, Israel).

Infrared (IR) spectra. IR spectra of the chemical products were taken as neat in NaCl prisms, and the unmodified and jojoba-modified membranes were taken as films on a Perkin-Elmer 357 spectrometer (Norwalk, CT).

[1]H Nuclear magnetic resonance (NMR) spectra. NMR spectra were run on a 200 MHz Bruker WP-200SY instrument (Spectrospin AG, Salladen, Switzerland) in CDCl₃.

Scanning electron microscope (SEM). Pictures were taken by JSM 35 CS model (JEOL Instrument, Tokyo, Japan).

Allylic bromination of jojoba wax. Allylic bromination of crude jojoba wax (I) was carried out with NBS in CCl₄, as previously reported (10). A molar ratio of 2:1 of NBS to jo-

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$(Z,Z) - CH_3(CH_2)_7CH=CH(CH_2)_mCOO(CH_2)_nCH=CH(CH_2)_7CH_3$

Jojoba wax

$\xrightarrow{2\,NBS}$ $CH_3(CH_2)_6[CHCH=CH(CH_2)_p]_2COO$
                          |
                          Br

J-2Br* (II)

$\xrightarrow{4\,NBS}$ $CH_3(CH_2)_6[CHCH=CHCH(CH_2)_{p-1}]_2COO$
                          | |
                          Br   Br

J-4Br* (III)

*one of several possible isomers, *p* = *m,n*.

SCHEME 2

joba wax was used for the synthesis of the allyl dibromo derivative of jojoba wax (J-2Br), and a 4:1 ratio for the production of allyl tetrabromojojoba (J-4Br) (Scheme 2).

Allylic amination of jojoba. Allylic amination of the bromo derivatives of jojoba was carried out with hexamethylene tetraamine as previously reported (11). ^{1}H NMR and IR spectra of the products (J-2NH$_2$ and J-4NH$_2$) were in accordance with published spectra (11).

Chlorosulfonation of PE. PE in film (20×20 cm) or hollow fiber (50×0.05 cm) form was purified before use by Soxhlet extraction in hexane for 24 h and then dried at room temperature. Thereafter, the hollow fiber or film was preswollen in dichloroethane (DCE), and introduced into the reactor (2 L) equipped with an inlet and outlet connection for gases. Chlorosulfonation was carried out at 65–70°C under ultraviolet (UV) illumination with a 2:1 ratio of SO$_2$ to Cl$_2$ (12). The volume ratio of the two gases was controlled by flowmeters. The chlorosulfonated product (PE-SO$_2$Cl, PESC) was cleaned by refluxing in a Soxhlet apparatus with a mixture of DCE and methanol (2:1) and dried in air overnight. The properties of the chlorosulfonated polymer were evaluated in terms of swelling in different solvents, ion-exchange capacity (after base hydrolysis to obtain the cation exchanger PE-SO$_3$H), and elemental analysis.

Amination of chlorosulfonated PE. Amination of PE-SO$_2$Cl was carried out as previously reported (13).

Binding of jojoba wax to PE. Chlorosulfonated PE (200 mg) was preswollen in solution of 20% J-xNH$_2$ in the appropriate solvent (dioxane, chloroform, or DMF) for 3 h at room temperature. The reaction mixture was maintained at 60°C for 24 h. The product (PE-SO$_2$-NH-J) was washed with the same solvent as used for preswelling and with chloroform (for removing unreacted jojoba) and dried under vacuum at room temperature. The amount of jojoba wax bound to polymer was calculated from differences in weights.

Reaction at the double bond in jojoba bonded to PE. To 200 mg of polymer PE-SO$_2$NH-J, preswollen in CCl$_4$, an equivalent amount (relative to the amount jojoba in the polymer) of Br$_2$ in CCl$_4$ was added dropwise over a period of 1 h. The mixture was allowed to stand for 2 h at room temperature, washed with CCl$_4$ and saturated NaHSO$_3$ solution, and finally dried under vacuum at room temperature. Bromine found: 19.2% (calc. 19.5%).

RESULTS AND DISCUSSION

Theoretically, the binding of jojoba wax to PE can be achieved in two different ways: (i) binding of allylic bromo derivatives of jojoba (J-xBr) to aminated PE (PE-SO$_2$NH~~~NH$_2$) or (ii) binding of allylic amino derivatives of jojoba (J-xNH$_2$) to chlorosulfonated PE (PE-SO$_2$Cl). For the first method, a variety of different diamines and polyamines were investigated, but all gave very low yields [3–8% (w/w) jojoba in polymer] (results not shown). However, the binding of jojoba to PE by the second method was effective, with yields reaching 98%. The amount of jojoba wax bound to polymer was in the range of 8–48% (w/w of the obtained polymer), depending on the reaction conditions.

Chlorosulfonation of PE. The properties of the three chlorosulfonated PE films (PESC series) are presented in Table 1.

Alongside chlorosulfonation, direct chlorination takes place as a competing reaction. The ratio between the two competing reactions depends on the proportion of gases and the temperature and duration of the reaction (12,14). Therefore, depending on the reaction conditions, we obtained a range of products with different contents of reactive groups (SO$_2$Cl and Cl). Calculations on the basis of elemental analysis showed that for every 9–10 units of ethylene in the polymer, there was one active SO$_2$Cl group:

TABLE 1
Characteristics of Chlorosulfonated Polyethylene[a]

Designation	S (%)	Cl (%)	mmol[b] S/g polymer	mmol[b] Cl/g polymer	S/Cl	Swelling (wt%) DMF	CHCl$_3$	Dioxane	IEC[c] (Q$_{H+}$ meq/g)
PE	0	0	0	0	—	1	4	9	0
PESC1	11.3	14.0	3.5	4.0	0.8	80	65	40	2.3
PESC2	8.7	18.9	2.7	5.7	0.5	79	60	38	2.2
PESC3	5.8	11.8	1.8	3.3	0.5	76	62	39	1.8

[a]PE = polyethylene, PESC = chlorosulfonated polyethylene.
[b]Content of element *X* in polymer (mmol/g) = *X*(%) · 1000/100 · MW.
[c]IEC = ion-exchange capacity.

$$[-CH_2-CH_2-]_{\{7-8\}}[-CH_2-CH-][-CH_2-CH-]$$
$$\quad\quad\quad\quad\quad\quad SO_2Cl \quad\quad Cl$$

Distribution of the functional groups in the polymer was examined by SEM (Figs. 1A and 1B). Distribution of the elements S and Cl in a cross-section of chlorosulfonated PE was

determined by means of the line scanning profile (LSP) of these elements in the polymer. Figure 1C shows homogeneous distribution of S and Cl in the polymer, indicating that the chlorosulfonation reaction proceeded not only on the surface, but also in the bulk, of the polymer; this facilitates further modifications inside the polymer, if so required. The swelling
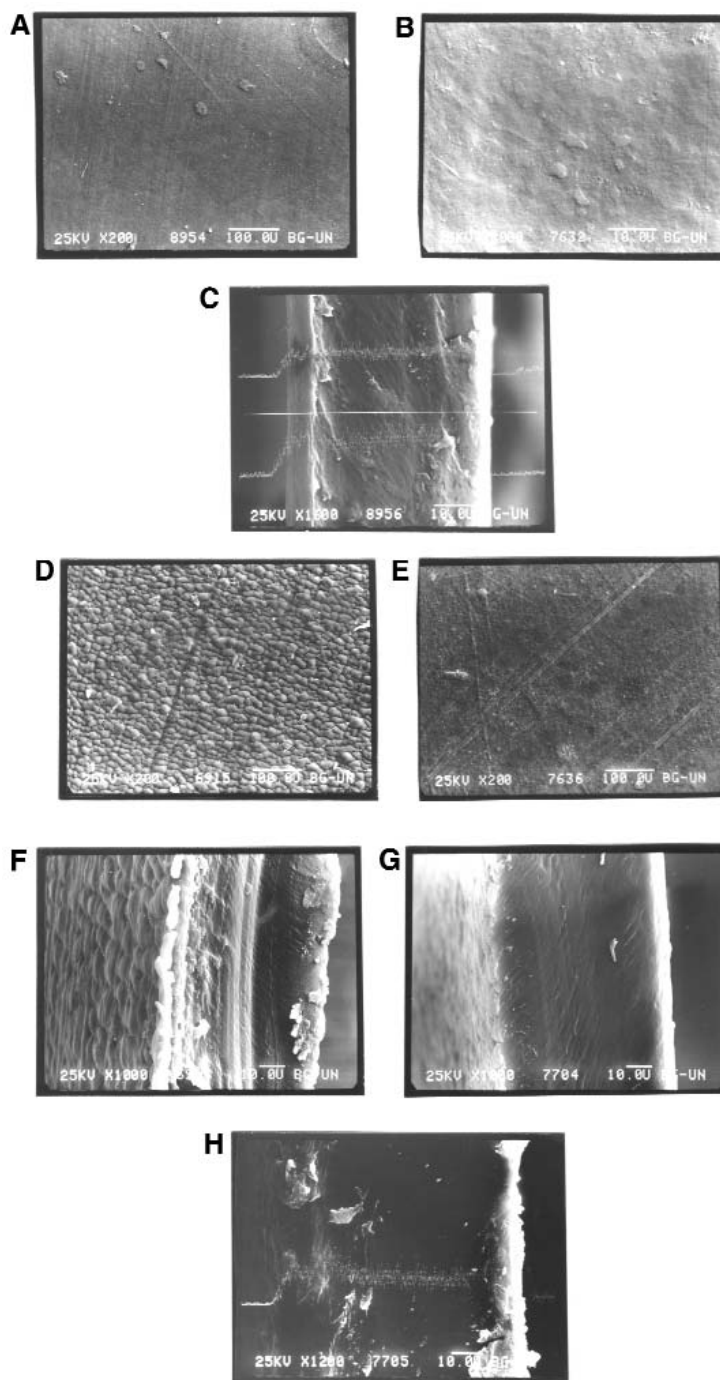
FIG. 1. Scanning electron micrographs of (A) polyethylene film; (B) chlorosulfonated polyethylene; (C) line scanning profile (LSP) of sulfur (upper line) and chlorine (lower line); (D) membrane surface after chemical binding of jojoba in dimethylformamide (J2NP1) (see Table 2); (E) membrane surface after chemical binding of jojoba in dioxane (J2NP2); (F) cross-section of J2NP1; (G) cross-section of J2NP2; (H) LSP of bromine in J2NP1 after bromination.

[CH$_3$(CH$_2$)$_6$CHCH=CH(CH$_2$)$_p$]$_2$COO
|
Br

J-2Br* (II)

↓ hexamine.HCl

[CH$_3$(CH$_2$)$_6$CHCH=CH(CH$_2$)$_p$]$_2$COO
|
NH$_2$

J-2NH$_2$* (IV)

[CH$_3$(CH$_2$)$_6$CHCH=CHCH(CH$_2$)$_{p-1}$]$_2$COO
| |
Br   Br

J-4Br* (III)

↓ hexamine.HCl

[CH$_3$(CH$_2$)$_6$CHCH=CHCH(CH$_2$)$_{p-1}$]$_2$COO
| |
NH$_2$ NH$_2$

J-4NH$_2$*(V)

*one of several possible isomers, *p = m,n.*
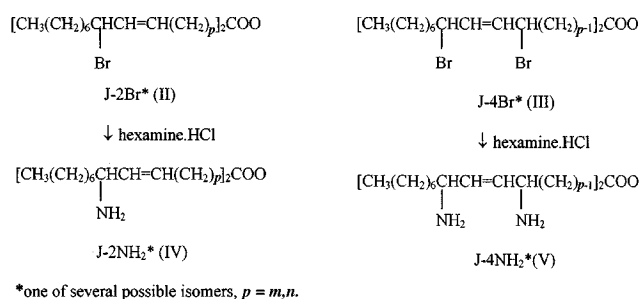
SCHEME 3

properties of the chlorosulfonated PE differ from solvent to solvent, and this property is the key factor in the chemical reaction of chlorosulfonated PE with allylic aminojojoba (see below): DMF was found to be the best solvent for the reaction.

Binding of jojoba wax to PE. Allyl aminated derivatives of jojoba (J-2NH$_2$ and J-4NH$_2$) were obtained by reacting the allyl brominated derivatives dibromojojoba (J-2Br) and tetrabromojojoba (J-4Br) with hexamine (Scheme 3).

The allylic amino jojoba derivatives were then reacted with chlorosulfonated PE by nucleophilic substitution to produce PE bound to jojoba *via* a stable sulfonamide bond. A series of reactions were performed under different conditions (type of solvent, percentage of jojoba in reaction mixture, and temperature) as shown in Table 2.

Inspection of the effect of the solvent showed that much less bonded jojoba was produced with dioxane as the solvent than with DMF or chloroform (Table 2, J2NP2 vs. J2NP1, J2NP4 vs. J2NP5 and J2NP8). These results are in keeping with the results for solvent uptake by chlorosulfonated PE (Table 1). An increase in temperature from 50 to 60°C did not have a significant influence on the weight increase in the product (product J2NP5 contained only a little more jojoba wax than J2NP6, or J2NP8 than J2NP9, in the same solvent).

However, a substantial drop in temperature did have a significant effect; for example, at room temperature, the binding of jojoba to PE did not succeed (product J2NP10). The concentrations of functional groups −SO$_2$Cl and −NH$_2$ also influenced the degree of binding of jojoba to the polymer (J2NP6 vs. J2NP7 for −NH$_2$, and J2NP1, J2NP5 and J2NP11 for −SO$_2$Cl, compare with Table 1). In general, the degree of the substitution depended on the reaction conditions. The total increase in weight of the starting PE-SO$_2$Cl film after J-2NH$_2$ bonding was found to cover a wide range of values, from 9 to 98%. The products were characterized by IR spectra, elemental analysis (Table 3), and SEM (Fig. 1).

Elemental analysis showed that the products did indeed contain jojoba (appearance of nitrogen of J-xNH$_2$, and loss of Cl in the product). Calculation showed that the amount of nitrogen introduced into the polymer was equivalent to the amount of Cl removed from the SO$_2$Cl group to form SO$_2$NHR. The yield of the reaction (% of reacted −SO$_2$Cl groups), calculated on the basis of elemental analysis, was significantly higher in DMF than in dioxane or chloroform (Table 3).

From the IR spectra of the products, it was clear that the polymer contained bonded jojoba. In addition to the characteristic bands of chlorosulfonated PE, i.e., absorption bands in the region of 1160–1170 and 1370 cm^{-1} attributed to the −SO$_2$Cl group (symmetric and asymmetric stretching) and the characteristic bands of the −CH group (broad peak in the region of 2800–2900 cm^{-1} and an intense band at 1460 cm^{-1}), new absorption bands appeared in the spectrum—of the carbonyl group of jojoba at 1740 cm^{-1}, a sulfonamide peak at 1686 cm^{-1}, and an amino group band in the range 3400–3500 cm^{-1}. These new peaks provided further evidence for bonding of the jojoba wax to the PE.

Nature of bonding of jojoba wax. A mechanism for the binding of allylic amino jojoba to chlorosulfonated PE can be proposed on the basis of the experimental observations. The allylic diamino jojoba derivative has two reactive amino

TABLE 2
Binding of Allylic Amino Jojoba Derivatives to Chlorosulfonated PE

Reagent[a]	Product	Solvent	Time of reaction (h)	J-xNH$_2$ in solution (%)	Temp. (°C)	Increase in weight (%)	Jojoba wax in product (%)
PESC1	J2NP1	DMF	24	20[b]	60	98	48
PESC1	J2NP2	Dioxane	24	20[b]	60	30	23
PESC2	J2NP3	Dioxane	24	10[b]	50	9	8
PESC2	J2NP4	Dioxane	24	20[b]	60	25	20
PESC2	J2NP5	DMF	24	20[b]	60	77	43
PESC2	J2NP6	DMF	24	20[b]	50	71	42
PESC2	J2NP7	DMF	24	10[b]	60	47	32
PESC2	J2NP8	Chloroform	24	20[b]	60	53	34
PESC2	J2NP9	Chloroform	24	20[b]	50	49	33
PESC2	J2NP10	DMF	24	20[b]	25	0	0
PESC3	J2NP11	DMF	24	20[b]	60	68	41
PESC3	J2NP12	Dioxane	24	20[b]	60	19	16
PESC2	J4NP1	DMF	24	10[c]	60	67	41
PESC2	J4NP2	Dioxane	24	10[c]	50	13	11

[a]For identification and abbreviation see Table 1. DMF, dimethyl formamide.
[b]x = 2.
[c]x = 4.

TABLE 3
Characterization of Products of Binding of Jojoba Wax to Chlorosulfonated PE

Product[a]	Solvent	Before reaction		After reaction			$-SO_2Cl$ reacted groups (%)
		S (%)	Cl (%)	S (%)	Cl (%)	N (%)	
J2NP1	DMF	11.3	14.0	5.2	1.8	2.8	86
J2NP2	Dioxane	11.3	14.0	8.1	8.0	1.4	34
J2NP5	DMF	8.7	18.9	4.7	7.0	2.1	82
J2NP8	Chloroform	8.7	18.9	ND	9.0	1.5	56
J2NP11	DMF	5.8	11.8	3.5	3.5	2.4	94
J2NP12	Dioxane	5.8	11.8	4.8	8.4	1.1	30
J4NP1	DMF	8.7	18.9	ND	2.9	2.3	100

[a]See Table 2; ND, not determined. See Tables 1 and 2 for abbreviations.

groups, one on each side of the ester group. During nucleophilic substitution on the chlorosulfonic group of PE-SO$_2$Cl, one of the diamino groups might bind to the polymer *via* a SO$_2$-N bond, and the second amino group might behave in one of two possible ways (Scheme 4): it may remain unreacted or it may take part in a nucleophilic substitution reaction to create an additional sulfonamide bond. Therefore, one molecule of jojoba wax can be bound at two sites along the polymer chain or it may be bound to two sulfonated PE chains (crosslinking).

The nature of bonding of J-2NH$_2$ to the chlorosulfonated PE—calculated on the basis of the analytical results given in Table 3—may be deduced from the data presented in Table 4. The results show that only in DMF did both mono- and di-amination reactions take place. The amount of bonded jojoba in this solvent was higher than that in dioxane or chloroform. Therefore, in products J2NP1, J2NP5, and J2NP11, jojoba wax was bonded at two sites to the polymer matrix, either, as shown in Scheme 4, to the same chain or, perhaps crosslinked to another polymer chain.

The nature of the bonding of the allylic tetraamino jojoba derivative to chlorosulfonated PE in the product J4NP1 (prepared in DMF) was also investigated by elemental analysis (Table 3). From the calculations, it appears that this derivative of jojoba bonded to the polymer by both single and double attachment but that the dominant mode was double attachment (Table 4).

Properties of the modified polymer. Some of the physicochemical properties of the new membranes prepared by bond-

ing of jojoba wax to chlorosulfonated PE *via* the stable sulfonamide bond were tested, i.e., electrical resistance, permselectivity, and ion exchange capacity (Table 5). Both membranes J2NP2 and J2NP1 (Table 2) showed relatively high hydrophobicity (Table 5), despite the fact that J2NP2 contained far fewer jojoba chains than J2NP1 and hence more unreacted $-SO_2Cl$ groups. The difference between the two membranes was shown by hydrolysis: J2NP1 and J2NP2 were hydrolyzed to yield products (J2NP1h and J2NP2h, respectively) that exhibited much higher hydrophilicity (lower electrical resistance and higher permselectivity, Table 5) than their predecessors, but J2NP2h, which contained more $-SO_3H$ groups, showed a greater similarity to hydrolyzed chlorosulfonated PE (PE-SO$_3$H) than J2NP1h. The same trend was evident for Q_{H+}: the ion-exchange capacity of membrane J2NP2h has much higher than that of J2NP1h.

The product of the reaction between allylic tetramino jojoba with chlorosulfonated PE (J4NP1) was much less hydrophobic than the products of reactions between allylic diamino derivatives of jojoba with PE-SO$_2$Cl (Table 5, lower electrical resistance and higher permselectivity than for J2NP1 and J2NP2). We assume that this phenomenon is related to the number of amino groups in the product. The tetraamino derivative (J-4NH$_2$) was more hydrophilic than J-2NH$_2$ (11) and, therefore, the resulting membrane (J4NP1), which contained more $-NH_2$ groups, was more hydrophilic than J2NP1.

Structure. SEM was used to determine the location of jojoba wax in the products. Pictures D and E in Figure 1 show
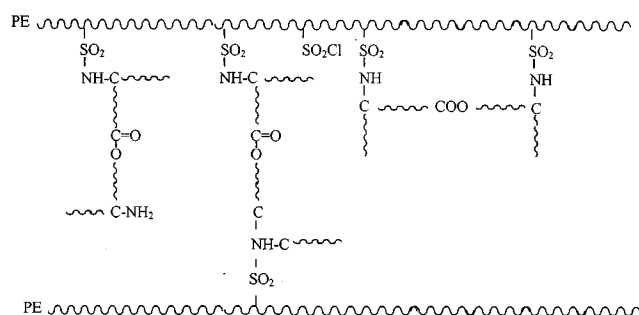
SCHEME 4

TABLE 4
Bonding of Allylic Diaminojojoba Derivatives to Chlorosulfonated PE

Product	Solvent	Amine attachment[a]		$-SO_2Cl$ reacted groups (%)
		Single	Double	
J2NP1	DMF	64	36	86
J2NP5	DMF	67	33	82
J2NP11	DMF	78	22	94
J2NP2	Dioxane	100	—	34
J2NP12	Dioxane	100	—	30
J2NP8	Chloroform	100	—	56
J4NP1	DMF	38	62	100

[a]Calculated value on the basis of mmol introduced amine/g and mmol reacted Cl/g. See Tables 1 and 2 for abbreviations.

TABLE 5
Properties of Modified PE

Membrane	Electrical resistance (Ω*cm^2)	Permselectivity[b] (mV)	IEC[c] Q_{H+} (meq/g)
PE-SO$_2$Cl	53942.9	0.2	—
J2NP1	36224.9	0.4	—
J2NP2	48619.1	0.3	—
J4NP1	2000.0	1.4	—
J2NP1h[a]	2435.5	2.0	0.01
J2NP2h[a]	185.5	8.6	0.33
PE-SO$_3$H	22.7	16.0	2.01

[a]Products of J2NP1 and J2NP2 after hydrolysis.
[b]Ref. 15.
[c]IEC, ion exchange capacity of membrane. See Tables 1 and 2 for other abbreviations.

the differences in the surfaces of products J2NP1 and J2NP2: for J2NP1, the polymer surface was covered with a thick layer of jojoba (picture D), whereas the surface of J2NP2 was similar to that of chlorosulfonated PE (picture E). Pictures F and G show cross-sections of the products J2NP1 and J2NP2. Because the content of jojoba wax in J2NP2 was low in comparison to that in J2NP1 [23% wax (w/w) per gram of J2NP2 vs. 48% in J2NP1] and jojoba bonded at one site in product J2NP2, it seems likely that jojoba formed a monolayer on the surface of the polymer rather than penetrating into the polymer matrix. In J2NP1, the jojoba content was high, and therefore, in addition to forming a monolayer on the surface, it was likely that a small amount of jojoba penetrated into the polymer and was distributed throughout the volume of the polymer. For testing this hypothesis and examining the reactivity of the double bonds in the bonded jojoba, J2NP1 was submitted to a bromination reaction.

Bromine addition to jojoba wax bound to PE. The degree of bromination, estimated by elemental analysis of bromine (see Experimental Procedures section), showed that the double bonds (DB) remained intact during the chemical binding of the wax to the chlorosulfonated PE (1.2 mmol DB/g gave 2.4 mmol Br/g). X-ray probe analysis of the distribution of Br in a cross-section of brominated J2NP1 (Fig. 1H) showed that Br was not distributed homogeneously along the inner section of the film: content of Br was higher at the edges of the film. This finding is in keeping with Picture D (Fig.1), which shows a thick layer of jojoba on the polymer surface, and some penetration into the bulk of the polymer.

The modified PE membranes and hollow fibers were tested for their suitability for application in separation processes, such as ion-exchange and pervaporation (16).

ACKNOWLEDGEMENT

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