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Advances in Radiation Processing of Polymeric Materials

Progrès réalisés en traitement par irradiation des polymères

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ADVANCES IN RADIATION PROCESSING OF POLYMERIC MATERIALS

by

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PROGRÈS RÉALISÉS EN TRAITEMENT PAR IRRADIATION DES POLYMÈRES

par

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RÉSUMÉ

Dans cette communication technique, nous examinons les progrès récents en applications industrielles de l'irradiation au faisceau d'électrons pour le traitement des polymères, au Takasaki Radiation Chemistry Research Establishment (TRCRE) de l'Institut de recherches sur l'énergie atomique du Japon (JAERI) et aux Laboratoires de Whiteshell d'EACL Recherche au Canada. L'irradiation d'un substrat à l'aide du rayonnement ionisant produit des radicaux libres par la formation d'espèces ionisées et excitées. On se sert des propriétés chimiques postérieures de ces radicaux dans le traitement par irradiation pour remplacer les techniques classiques de traitement basées sur le chauffage et/ou l'ajout de produits chimiques. Les avantages du traitement par irradiation comprennent la création de nouveaux produits possédant les propriétés matérielles recherchées, une rentabilité globale de procédé et, souvent, des avantages du point de vue de l'environnement.

À EACL Recherche, on mène des études sur des complexes renforcés de fibres de carbone et d'aramide pour une variété d'applications structurelles. Ces complexes, utilisés surtout dans l'industrie aérospatiale, sont produits normalement par le traitement thermique d'époxydes. La production à la température ambiante de matrices équivalentes d'époxy acrylaté, au moyen du traitement par irradiation, peut servir à créer des complexes ayant des contraintes résiduelles réduites et ne donnant lieu à aucune émission importante de composés volatils. La dégradation par irradiation des matières à la suite d'une rupture de liaison peut être exploitée pour la fabrication de la pâte à bois et de la viscose. À EACL Recherche, des études du traitement par irradiation de copeaux de bois avant leur réduction en pâte on montré que, dans certains cas, l'irradiation préalable des copeaux de bois facilite le procédé de réduction en pâte, ce qui a pour effet de réduire de façon importante la consommation totale d'énergie, sans incidence importante sur la qualité de la pâte. En outre, des études menées à EACL Recherche ont montré que le traitement par irradiation peut servir à produire de meilleurs thermoplastiques à fibre de bois et à charge de poudre minérale pour diverses applications.

Au TRCRE, où on étudie présentement les propriétés mécaniques de couches traitées au faisceau d'électrons de mélanges d'oligomères aliphatiques uréthamiques acryliques et de monomères à une seule ou à plusieurs fonctions, on a étudié le traitement de mélanges d'oligomères et de monomères à l'aide de faisceaux d'électrons à faible énergie. Ces études ont mené à la création d'une série d'oligomères uréthanniques acryliques qui conviennent aux adhésifs sensibles à la pression. Enfin, une application nouvelle du rayonnement ionisant, à l'étude au TRCRE, consiste à greffer des groupes fonctionnels sur des substrats polymères dans le but de produire des matières ayant les propriétés physico-chimiques voulues. Par exemple, les séparateurs d'accumulateurs sont préparés en greffant des monomères d'acide acrylique sur des substrats de polyéthylène; de plus, des adsorbants d'ammoniaque et d'amines ont été créés en greffant du p-styrène sulfonate de sodium et de l'acide acrylique sur des étoffes de nappes de fibres.

Cette communication technique a été présentée au Congrès nucléaire international de l'ANC/la SNC (CNI '93) à Toronto (Ontario) en 1993.

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ABSTRACT

In this paper we review recent advances in industrial applications of electron-beam irradiation in the field of polymer processing at the Takasaki Radiation Chemistry Research Establishment (TRCRE) of JAERI (Japan Atomic Energy Research Institute), and the Whiteshell Laboratories of AECL Research, Canada. Irradiation of a substrate with ionizing radiation produces free radicals through ionization and excitation events. The subsequent chemistry of these radicals is used in radiation processing as a substitute for conventional processing techniques based on heating and/or the addition of chemicals. The advantages of radiation processing include the formation of novel products with desirable material properties, favourable overall process economics and, often, environmental benefits.

At AECL Research, carbon and aramid fibre-reinforced composites are being studied for a variety of structural applications. These composites, primarily used in the aerospace industry, are normally produced by thermal curing of epoxies. Ambient temperature production by radiation processing of equivalent acrylated epoxy matrices can be used to produce composites with reduced residual stresses and without any substantial emission of volatile compounds. Radiation-induced degradation of materials as a result of bond scission can be exploited to process pulp and viscose. At AECL Research, studies of radiation processing of wood chips prior to pulping have shown that, in certain cases, pre-irradiation of the wood chips facilitates the pulping process, resulting in a significant decrease in the overall energy required without any significant impact on the quality of the pulp. Also at AECL Research, studies have shown that radiation processing can be employed to produce better wood-fibre and mineral-powder-filled thermoplastics for a variety of applications.

Curing of oligomer/monomer mixtures with low-energy electron beams (EB) has been studied at TRCRE, where the mechanical properties of EB-cured films of mixtures composed of aliphatic urethane-acrylate oligomers and mono- or multi-functional monomers are being studied. These studies led to the development of a series of urethane acrylate-type oligomers suitable for pressure-sensitive adhesives. Finally, a novel application of ionizing radiation, studied at TRCRE, involves grafting functional groups onto polymeric substrates to produce materials with desirable physicochemical properties. For example, battery separators are prepared by grafting acrylic acid monomers onto polyethylene substrates; also adsorbents for ammonia and amines have been developed by grafting sodium p-styrenesulfonate and acrylic acid onto non-woven fabrics.

This paper was presented at the CNA/CNS International Nuclear Congress (INC '93), held in Toronto, ON, 1993.

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1. INTRODUCTION

In this paper we review recent advances in industrial applications of ionizing radiation, primarily electron beams, in the field of polymer processing at two leading laboratories in this field, the Takasaki Radiation Chemistry Research Establishment (TRCRE) of JAERI (Japan Atomic Energy Research Institute), and the Whiteshell Laboratories of AECL Research, Canada.

The Radiation Applications Research program of AECL Research was established in 1985 to research and develop new industrial applications of high-energy electrons, made possible by AECL's 50-kW, 10-MeV IMPELA accelerator. The program currently employs about 15 researchers and is focussing on materials processing, particularly curing polymers and advanced composites, and investigating biomass applications. The research presented here was performed with AECL's I-10/1 research and pilot production electron accelerator, which is a 1-kW, 10-MeV prototype.

TRCRE was founded in 1963 as a centre for research and development of radiation chemistry. Currently, the centre has three electron accelerators: a 3-MeV, 25-mA Dynamitron; a dual-beam 2-MeV, 30-mA Cockcroft-Walton-type accelerator (made by Nissin High Voltage (NHV)); and a 300-keV, 100-mA Curetron. The two medium-energy accelerators are being used mainly for R&D work on crosslinking and other irradiation effects on polymers, on grafting onto polymers, as well as for pollution control studies. The low-energy accelerator was installed in 1985 and has been used mainly for curing liquid resins.

Irradiation of a substrate with ionizing radiation produces ions and free radicals through ionization and excitation events. The subsequent chemistry of these radicals is used in radiation processing to substitute for conventional processing techniques based on heating and/or the addition of chemicals (1-4). The advantages of radiation processing include the formation of novel products with desirable material properties, such as increased strength and hardness, higher melting temperature, and reduced residual stress; favourable overall process economics, such as increased processing efficiency or reduced energy requirements; and often environmental benefits, such as the generation of reduced amounts of volatile compounds or lower consumption of aggressive chemicals.

Crosslinking of polyethylene, polyolefins, and elastomers represent typical radiation processing applications that can be used, for example, in the production of wire and cable insulation, heat-shrinkable tubing and film, plastic foams, and tires [1,2]. New radiation applications under study are reviewed below.

2. APPLICATIONS

2.1 ADVANCED COMPOSITES

At AECL Research, carbon and aramid fibre-reinforced composites are being studied for a variety of structural applications, because of their high

strength-to-weight ratio and corrosion resistance. These composites, primarily used in the aerospace industry, are normally produced by thermal curing of epoxies. However, thermal curing is time-consuming, generally produces toxic gaseous emissions, and can lead to residual stresses in the product as a result of a mismatch between the thermal expansion coefficients of the fibres and the epoxy matrix. Ambient temperature production by radiation processing of equivalent acrylated epoxy matrices can be used to produce composites with reduced residual stresses, and without any substantial emission of volatile compounds [3,4]. In addition, production of such composites by continuous radiation processing is expected to be one to two orders of magnitude (minutes rather than hours) faster than by batch-type thermal processes [3,5,6].

Most epoxy formulations have been developed for conventional thermal curing techniques. Such epoxies polymerize by electron processing via cationic mechanisms, which are inhibited by traces of moisture; thus, electron processing becomes impractical. To circumvent this problem the epoxy oligomer is acrylated [3]. As shown in Table 1, tests of matrix materials for advanced composites required by the aerospace industry demonstrate that such acrylated epoxies, cured by electron processing, are equivalent to thermally cured conventional epoxies [3,5,7]. Current studies with acrylated epoxy composites reinforced with aramid fibres show compression strengths below the acceptable values [8]. However, we expect that the properties of this system can also be improved by a suitable choice of matrix resin and fibre/matrix coupling agents.

TABLE 1

TYPICAL PROPERTIES OF CARBON-FABRIC/EPOXY LAMINATES

Material[1]	Tensile Properties			Compression Properties		
	Strength (MPa)	Modulus (GPa)	Strain (%)	Strength (MPa)	Modulus (GPa)	Strain (%)
EB-cured epoxy[2]	580	57	1.1	460[3]	30	0.7
Thermally cured epoxy	460	57	0.8	460	50	0.9

[1] 14-ply, 600-kPa curing pressure, plain-weave AS4 fabric, 64% fibre volume.
[2] Acrylated epoxy.
[3] C.B. Saunders, V.J. Lopata, T.E. McDougall, W. Kremers and A. Singh, unpublished data.

In Table 2 we present data on residual stresses, obtained by radius of curvature measurements (the higher the radius of curvature the lower the stress). These data show that electron-processed acrylated epoxy composites exhibit lower residual stresses than thermally treated epoxy composites [6].

TABLE 2

RADIUS OF CURVATURE (m) FOR DETERMINING RESIDUAL STRESSES OF SELECTED COMPOSITES [6]

Resin Blend	Electron-Cured Samples 0,90[1]	0,45[1]
EA-1[2],20% RH	0.41 ± 0.02	1.10 ± 0.17
EA-2, 20% RH	0.24 ± 0.01	0.64 ± 0.04
	Thermally Cured Samples	
Resin A[3]		
0% RH	0.06 ± 0.004	0.23 ± 0.020
65% RH	0.15 ± 0.002	0.59 ± 0.060
Resin B[4]		
0% RH	0.07 ± 0.007	0.21 ± 0.013
65% RH	0.14 ± 0.009	0.40 ± 0.030

[1] Two lay-up configurations (0, 90° and 0, +45°, -45°, -45°, +45°)
[2] EA: Commercially available epoxy acrylate formulations; RH, relative humidity
[3] Epoxy resin cured at 170°C for 1 h (2 MPa); post-cured at 190°C for 4 h.
[4] Epoxy resin cured at 140°C for 30 min; 170°C for 1 h (0.7 MPa).

2.2 POLYMERIC FILMS AND COATINGS

Curing oligomer/monomer mixtures with low-energy electron beams (EB) has been studied at TRCRE. A systematic study [9] of the mechanical properties of EB-cured films of various mixtures, composed of aliphatic urethane-acrylate oligomers and mono- or multi-functional monomers, established that mono-functional monomer mixtures can cover a wide range of tensile properties, giving higher values of both tensile strength and elongation than multi-functional monomers, as shown by the data in Table 3. It was also found that in the mono-functional monomer systems the tensile properties are dependent on the glass transition temperature (Tg) of the bulk polymer rather than that of the monomer. Some of these combinations with high Young's moduli also exhibit heat shrinkable properties.

The factors affecting adhesion properties of EB-cured coatings onto galvanized or primer-coated steel sheets have also been studied [10]. This study showed that an oligomer-monomer mixture with a polar component gives a coating with better adhesion to galvanized steel sheet, whereas a mixture

without any polar component showed better adhesion to the primer-coated steel. This study contributed to the industrial application of EB curing to the production of precoated steel coils by a Japanese steel company.

EB-curable oligomers have been developed using dimer acids, which are produced by dimerization of unsaturated higher fatty acids. A study on diacrylate oligomers of epoxidized dimer acids showed their potential application in coatings, preferably as filler coatings on wood panels [11].

TABLE 3

TENSILE PROPERTIES OF EB-CURED FILMS OF THE UX4101 OLIGOMER, WITH OR WITHOUT MONOMERIC ADDITIVES

Monomeric additives	Strength (kg/cm^2)	Elongation (%)	Modulus (kg/cm^2)
None (mono-func.)	170	140	20
Cyclohexyl acrylate[1]	300	240	70
Isobornyl acrylate[1]	410	160	5 700
Acrylic acid[1]	680	150	12 400
N-vinyl pyrollidone[2] (multi-func.)	560	110	12 000
1,6-hexane diacrylate[2]	180	30	1 500
NGTD[2,3]	320	30	9 100
PTPT[2,4]	240	70	270

[1] Oligomer/mono-functional monomer = 60/40
[2] Oligomer/multi-functional monomer = 70/30
[3] NGTD: Neopentylglycolated trimethylolpropane diacrylate
[4] PTPT: Propoxylated trimethylolpropane triacrylate)

Furthermore, dimer acid-based polyesters were converted to urethane di-acrylates (UDA). EB curing of UDA and UDA/monomer mixtures provided, in most cases, soft or elastic films with possible application to pressure-sensitive adhesives. Table 4 shows dose-to-cure and adhesive properties of UDA oligomers with different molecular weights. As the molecular weight increases, the dose-to-cure and adhesion strength increase. These studies also indicated that a wide range of adhesive properties can be obtained from UDA type of oligomers by changing the urethane acrylate compound and selecting the monomer(s) to be mixed.

TABLE 4

EFFECT OF MOLECULAR WEIGHT OF UDA ON DOSE-TO-CURE AND ADHESIVE PROPERTIES

Molecular Weight	6100	17 500	21 200	28 600
Dose (kGy)	10	20	100	100
Peel Strength[1] (g/inch)	3	400	600	1500
Ball Tack (No.)	<2	5	8	15
Holding Time[2] (h)	>24	>24	>24	4

[1] Stainless Steel
[2] 40°C, 1 kg

2.3 BIOMASS APPLICATIONS

Radiation-induced degradation of materials, as a result of bond scission, can be exploited to process pulp and viscose. At AECL Research, studies of radiation processing of wood chips prior to pulping have shown that, in certain cases, pre-irradiation of the wood chips facilitates the pulping process, resulting in a significant decrease in the overall energy required, with only minor impact on the quality of the pulp [12]. Furthermore, because irradiation-based pulping processes are expected to use fewer and less aggressive chemicals than conventional processes for producing pulp and cellulosic fibres, it offers significant environmental advantages.

Recent studies by AECL and Sunds Defibrator have shown that EB pretreatment of wood chips produces chemical changes in the wood resulting in net electrical power savings of 20 to 40% in the refiner mechanical pulping process [12]. These studies examined the effects of EB pretreatment on black spruce, loblolly pine and aspen wood chips. Although some reduction in intrinsic fibre strength and fibre length was noted, the specific energy savings are encouraging. The EB pretreatments were conducted in an air atmosphere, at room temperature, using AECL's I-10/1 electron accelerator. The treated chips were then packed in drums and air-freighted to the Sunds pulping pilot plant in Sundsvall, Sweden, for refining. The resulting power savings translate into substantial economic benefits. For example, for a mill producing thermomechanical pulp for newsprint from black spruce chips, where energy requirements can be up to 2200 kWh/t, a 25% reduction in electrical energy consumption would amount to annual savings exceeding $4.5 M.

Exposure of pulp to high-energy electrons or gamma rays can be used to reduce the degree of polymerization of cellulose [13-15]. Typical results using 10-MeV electrons and ^{60}Co gamma rays are shown in Figure 1. This treatment has been viewed as an efficient replacement of the "aging" step in viscose production. Furthermore, electron-treated pulp has been found to possess higher reactivity, resulting in decreased amounts of chemicals, such as CS_2, required in viscose production [15]. Also, decreased usage of CS_2 did not have any adverse effects on the properties of fibres produced

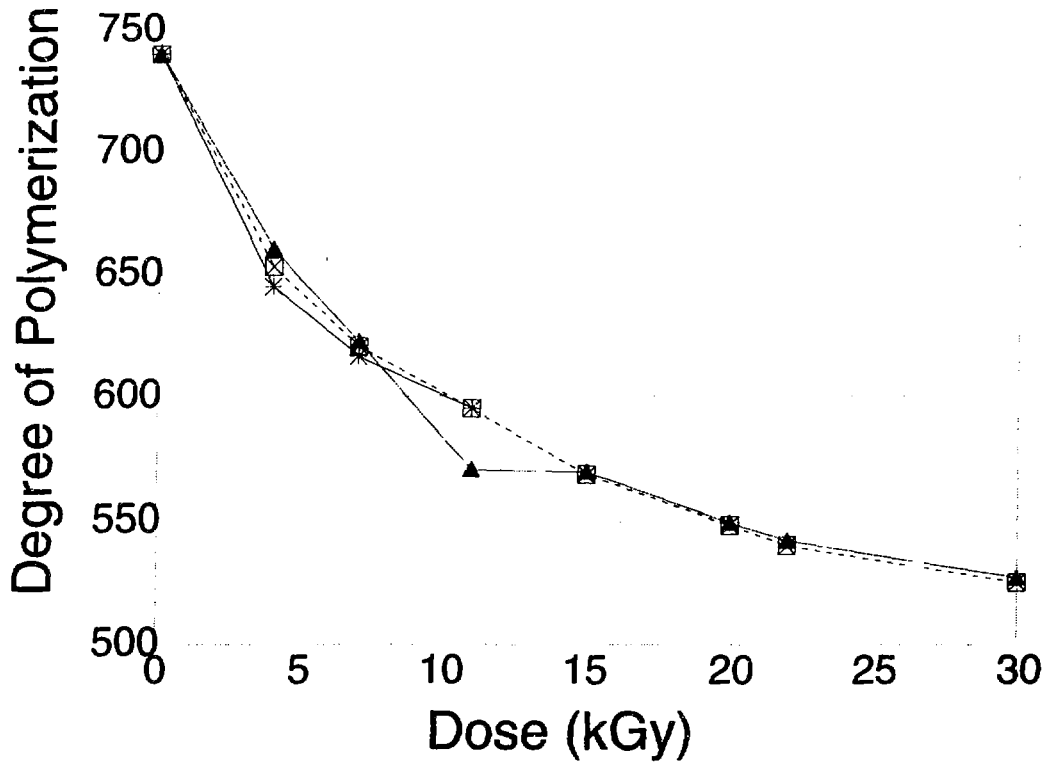

FIGURE 1: Effect of Electron (□ , * 10 MeV) and Gamma (▲ 1.3 MeV) Irradiation on Depolymerization of Cellulose

from viscose made with irradiated pulp. The benefits realized from this enhanced reactivity include both economic savings, in terms of lower chemical (CS_2, NaOH and H_2SO_4) usage and reduced environmental releases (CS_2 and H_2S).

Radiation-induced processing of natural and artificial polymers, such as wood-fibre-reinforced polypropylene (WFRP), yields a variety of products that have superior properties and conserve a non-renewable hydrocarbon resource (7). To make WFRP, wood fibre is mixed with polypropylene at the compounding stage, where proprietary reactive additives are also added to facilitate fibre-polypropylene chemical bond formation. The mixture is irradiated with the 10-MeV electron accelerator to a dose of 10 kGy and is then extruded to produce pellets. These pellets can be injection-moulded to fabricate desired products. As shown in Table 5, improvements in the properties of the irradiated material include higher heat deflection temperature, higher flexural modulus and higher flexural strength, compared with the wood-fibre-filled product. Similar improvements have also been obtained with the mineral powder/polypropylene system.

TABLE 5

COMPARISON OF PROPERTIES OF WOOD-FIBRE-FILLED AND REINFORCED POLYPROPYLENE

Composition/Properties	PP[1]	Filled[2]	WFRP[3]
Polypropylene (mass %)	100	65	65
Wood (mass %)	-	35	35
Tensile Strength (MPa)	37.3	35.3	48.8
Tensile Modulus (GPa)	1.87	3.93	3.89
Flexural Strength (MPa)	37.8	44.0	62.5
Flexural Modulus (GPa)	1.41	3.04	3.46
Impact Strength, notched (J/m)	12	11	11
Thermal Expan. Coeff. (10^6/°C)	134	23	18
Heat Deflection Temp. (°C)	66	94	116
Melt Flow Index (g/10 min)	10	0.5	4.4

[1] PP - polypropylene
[2] Filled, wood-fibre-filled PP
[3] WFRP, wood-fibre-reinforced PP (electron-processed)

2.4 OTHER APPLICATIONS

A novel application of ionizing radiation, studied at TRCRE, involves grafting functional groups onto polymeric substrates to produce materials with desirable physicochemical properties for a variety of applications (16). Table 6 summarizes R&D work on radiation grafting at TRCRE.

The application of electron beams to the sterilization of medical products is an emerging application worldwide. The radiation effects on microorganisms and the durability of polymeric materials used in medical ware have been reviewed recently [17]. The degradation of polypropylene, one of the main materials used in medical products, during EB irradiation and its storage after the irradiation have also been extensively studied at TRCRE. It was confirmed that degradation after EB irradiation is much less than after gamma irradiation. Chemiluminescence measurements of films showed that EB irradiation produces less oxidation and the depth of the oxidized layer is thinner than with gamma irradiation [18]. Further studies of the degradation of polypropylene showed that increasing the amorphous phase, either by using copolymers or polypropylene with higher molecular weights or by excluding nucleating agents, suppresses degradation of EB-treated polypropylene [19,20].

TABLE 6

R&D WORK ON RADIATION GRAFTING CARRIED OUT AT TRCRE

Substrates	Monomers	Treatment and Functional Groups	Use and Characteristics
Polyethylene Film (25 μm)	Acrylic Acid	Alkali Treatment; COOK or COONa	Battery Separators, Ion-Exchange Membranes, Long Durability, Low Electric Resistance
Fibrous or Nonwoven Polypropylene (1~20 μmϕ)	Acrylonitrile	Amidoximation with Hydroxyamine	Adsorbents for Uranium From Seawater and Heavy Metal Ions
Nonwoven Polypropylene (1~20 μmϕ)	Styrene	Sulfonation; $-SO_3H$	Adsorbents for Toxic Gases, such as NH_3, H_2S
Polyethylene Films (25 μm)	Acrylic Acid	Alkali Treatment; -COOK Radiation Crosslinking	Pervaporation Membranes for Alcohol vs. Water
Polyethylene Foam	Vinyl Phosphonate		Flame Retardant Polyethylene Foam
Polyethylene Hollow Fiber (id = 0.62 mm)	Glycidyl Methacrylate	Iminodiacetic Acid	Functionalized Hollow Fibre Membrane

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