PMMA Blended and DPNR-*g*-PMMA Coated DPNR and NR-LA for Dipping Applications

S. Kovuttikulrangsie,[1] K. Sahakaro,[1] C. Intarakong,[1] P. Klinpituksa[2]

[1]*Department of Rubber Technology and Polymer Science, Prince of Songkla University, Pattani, Thailand, 94000*
[2]*Department of Science, Prince of Songkla University, Pattani, Thailand, 94000*

ABSTRACT: Deproteinized natural rubber (DPNR) latex, prepared by digesting the protein within low-ammonia preserved concentrated (NR-LA) latex with alcalase enzyme at 40°C for 24 h, has the nitrogen content of 0.039 wt %. Poly(methyl methacrylate) (PMMA) and graft copolymers (DPNR-*g*-PMMA) latices were synthesized by emulsion polymerization technique for blending or coating on DPNR or NR-LA. Their general properties of natural and synthesized latices including compounded latex were verified. The friction coefficient of a homogenous film obtained from PMMA blended DPNR or NR-LA latex and a DPNR-*g*-PMMA coated DPNR and NR-LA film was found to be lower than those of films prepared by using DPNR or NR-LA latex. Furthermore, the DPNR-*g*-PMMA coated films had lower friction coefficient than those of PMMA blended films. The physical properties in terms of 300% modulus, tensile strength, and elongation at break of those rubbers were tested and confined to the ASTM 3377-78a and ASTM 3378-99 standards for dipping products of surgical gloves and examination gloves, respectively. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 833–844, 2004

Key words: rubber; latices; graft copolymers; polymerization; coating; blending

INTRODUCTION

Cis-polyisoprene natural rubber (NR) is an unsaturated elastomer with an electron-donating methyl group attached to carbon double bond in the main chain, which can facilitate a certain type of chemical modification. A chemical reaction, which is taken place directly on NR latex or on dry rubber, can be influenced by the presence of nonrubber components such as proteins. These nonrubber constituents can act as catalyst inhibitors while also influencing the process of free-radical reaction.[1] Proteins remaining in products made of NR latex are potential sensitizers. Therefore, reduction of protein levels in manufactured NR latex products is important for preventing sensitization and adverse allergic reactions to latex.[2] Modification of NR while still in latex form[3,4] was studied and employed industrially for more than 40 years. Most commercial processes for grafting monomers onto NR are based on redox polymerization.[5,6] A redox initiating system consisting of *tert*-butyl hydroperoxide (*tert*-BuHP) and tetraethylene pentamine (TEPA) was effectively used for the emulsion polymerization in NR latex. It is not sensitive to oxygen, although functioned well when ammonia was presented.[7] A surfactant (usually fatty acid soap) is used to stabilize the latex particle from coagulation. The deproteinized natural rubber grafted poly(methyl methacrylate) (DPNR-*g*-PMMA) latex is known as *Heveaplus*.[8] In this work, PMMA latex and graft copolymers (DPNR-*g*-PMMA) were synthesized by an emulsion polymerization technique for blending or coating on DPNR or natural rubber low ammonia (NR-LA). The homogenous vulcanized rubber films obtained from different weight fractions of PMMA in DPNR or NR-LA latex and the coated film of DPNR-*g*-PMMA were examined for their physical properties.

EXPERIMENTAL

Materials

Sixty percent NR-LA latex (produced by Patex Pattani Industry Co. Ltd., Thailand) was used to produce the deproteinized natural rubber, DPNR. Alcalase enzyme (Kao Co. Ltd., Japan) was used to digest the proteins in NR-LA latex. Sodium dodecyl sulfate (SDS) was used as a surfactant during the process of centrifugation. The monomer used for synthesis and grafting on DPNR latex was methyl methacrylate (MMA; Merck, Germany). The redox initiators were *tert*-butyl hydroperoxide solution (Fluka, Switzerland) and tetraethylene pentamine (Fluka). The emulsifier used in emulsion polymerization technique was an
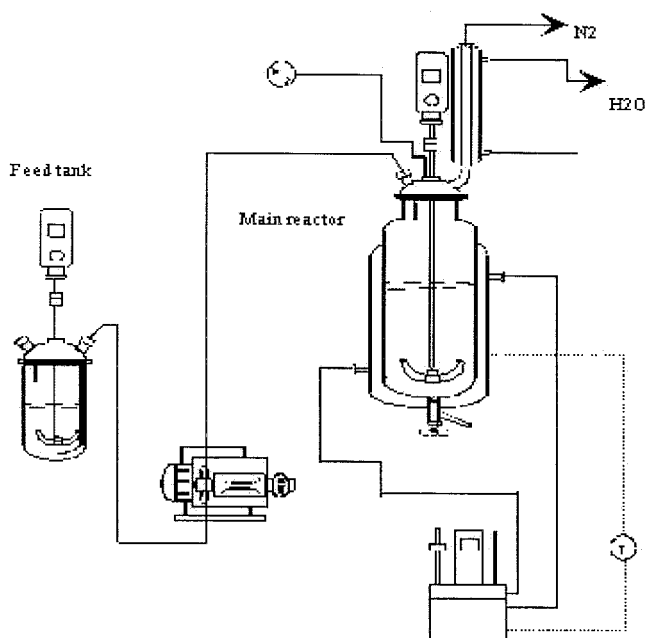
Figure 1 A schematic reactor system.

TABLE I
The Properties of DPNR and NR-LA Latices

Properties	DPNR latex	NR-LA latex
Dry rubber content (%)	60.32	60.36
Total solids content (%)	60.74	61.45
Alkalinity (%)	0.155	0.296
pH	9.77	10.44
Mechanical stability (s)	1,860	1,800
Surface tension (dyn/cm)	34.1	37.5
Nitrogen content (wt %)	0.039	0.408
Average particle size (μm)	0.83	1.10
Molecular weight (g/mol)	6.02×10^5	6.76×10^5

anionic-type surfactant, ammonium nonylphenylether sulfate (AD-33; SEPPIC, France). The ungrafted PMMA and NR present in the graft copolymer were extracted by using acetone and petroleum ether, respectively. The 50% zinc oxide dispersion used as activator was prepared by ball milling a mixture of zinc oxide, vultamol, bentonite, and distilled water for 48 h. The 50% antioxidant dispersion consisting of vultamol and distilled water was prepared by ball milling for 24 h. The 50% dispersion of vulcanizing agent was also prepared by ball milling a mixture of sulfur (Siam Chemical, Thailand), zinc diethyldithiocarbamate (ZDEC) (Vulnax, France), zinc mercapto-

benzothiazole (ZMBT) (Vulnax), vultamol (BASF, Germany), and water for 72 h.

Preparation of deproteinized natural rubber latex

NR-LA concentrated latex with 60% dry rubber content (DRC) was diluted to 30% DRC by using SDS (1% w/v) before incubating with 0.04% w/w of alcalase enzyme at 40°C while slowly stirring for 24 h. The reacted latex was controlled by washing twice to make 60% DRC latex using a centrifuge (an Alfa Laval model 410) at a speed of 10,000 rpm for 30 min. The surfactant, SDS (1% w/v), was added to obtain the required concentration. The average particle size of latex was analyzed by using a Coulter LS 230 analyzer and the nitrogen content of DPNR was analyzed by using the Kjeldahl method, according to ASTM D3533-90.

Preparation of poly(methyl methacrylate)

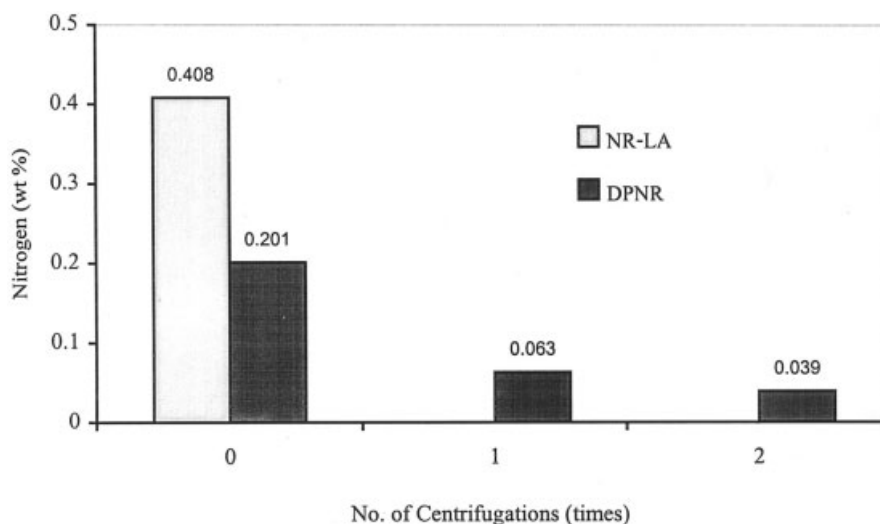A semicontinuous emulsion polymerization technique was used for this synthesis. Preemulsion was pre-

Figure 2 Nitrogen contents in NR-LA and DPNR.

TABLE II
The Properties of DPNR and NR-LA Latex Compounds

Properties	DPNR compounded latex	NR-LA compounded latex
Total solids content (%)	49.8	50.2
Surface tension (dyn/cm)	32.4	35.7
Viscosity (cP)	48.5	52.2
pH	10.2	10.5

pared in a feed tank consisting of MMA monomer (100 wt %), surfactant (2.68 wt %), $K_2S_2O_8$ (0.21 wt %), and $NaHCO_3$ (0.25 wt %). The main reactor initially consisted of surfactant (0.036 wt %) and $K_2S_2O_8$ (0.05 wt %). Deionized water was added to adjust the total solids content to about 40%. The components were agitated by means of a mechanical stirrer at a speed of 150 rpm at 70°C. The system was purged by nitrogen gas to deoxygenate the reactor for at least 15 min before adding the preemulsion from the feed tank to the main reactor with a flow rate of about 3 mm/min. After terminal feeding all ingredients, the temperature was increased to 80–85°C and the reaction was left to complete for 1.5 h.

Preparation of graft copolymer

Graft copolymer of deproteinized natural rubber and methyl methacrylate (DPNR-g-PMMA) was synthesized by using a semicontinuous emulsion polymerization technique (Fig. 1). The bipolar redox initiating system of $tert$-butyl hydroperoxide/tetraethylene pentamine was employed in the ratio of 1/1 based on the monomer, MMA. The MMA monomer contained 1.5 wt % of TEPA and 1.5 wt % of $tert$-BuHP. The surfac-

tant, AD-33, was about 1 wt % based on monomer weight. The feed tank was filled with a preemulsion containing MMA. The total amounts of the ingredients used for polymerization were always the same. Distilled water was added to produce the solids content of about 40%.

A 60% DPNR latex was initially charged into a main reactor (capacity of 2 L) together with 1.5 wt % TEPA. Ammonium solution (1 wt %) based on the DPNR latex was also added to maintain the pH (above 10.5) before starting the reaction. The components were then agitated by means of a mechanical stirrer at a speed of 150 rpm at 50°C. The system was purged by using nitrogen gas to deoxygenate the reactor for at least 15 min before adding the monomer ingredient from the feed tank to the main reactor with a flow rate of about 3 mm/min. After terminal feeding all ingredients, the temperature was increased to 60°C and the grafting reaction was left to complete for 1.5 h.
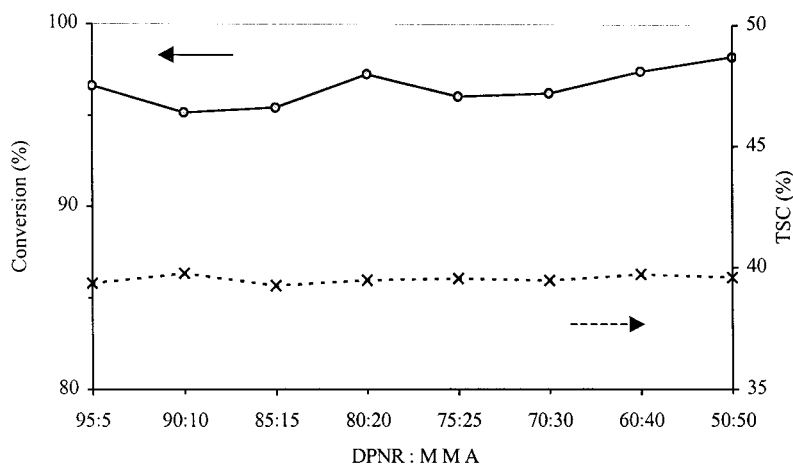
Latex compounds

The ingredients for latex compounds were as follows: 60% DPNR or LA-NR latex (167.0 g), 50% ZnO (2.0 g), 50% sulfur (3.5 g), 50% ZDEC (1.5 g), 50% ZMBT (0.5 g), 20% oleate soap (3.0 g), 50% $tert.$ butylhydroxy toluol (BHT) (2.0 g), 10% KOH (2.0 g), and H_2O (30.5 g). Rubber film was prepared by using a coagulant dipping method. Both latex compounds of DPNR and NR-LA were maturated to achieve the chloroform no. 2-3 before dipping.

Dipping process

The mold used for the dipping process was cleaned and dried before coating with 10% calcium chloride. A
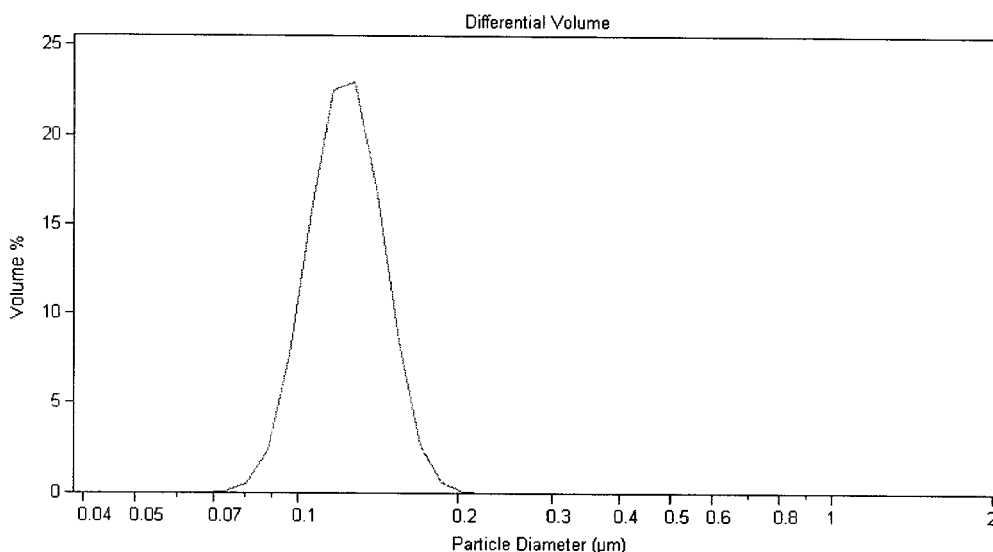
Figure 3 Particle size distribution curve of poly(methyl methacrylate) latex.

Figure 4 % Conversion and total solids content of the synthesized graft copolymer latex.

film was prepared by slowly dipping the mold into DPNR or NR-LA latex compounds for 3 s and drying at 60°C for 15 min before either vulcanizing at 110°C for 30 min or coating that rubber film with DPNR-*g*-PMMA latex before being vulcanized.

RESULTS AND DISCUSSION

DPNR or NR-LA latex properties

Nitrogen contents of DPNR (Fig. 2) were 50% decreased after digesting NR-LA latex with alcalase enzyme. After being centrifuged one and two times, the nitrogen content of DPNR latex was reduced to 0.063 and 0.039 (84 and 90% reductions), respectively.

The creaming latex particles obtained after centrifugation were 70–72% dry rubber content (DRC). The latex properties were shown in Table I. The dried rubber content of DPNR or NR-LA latex was then diluted to 60% with 0.1% w/w SDS. The total solids

content (TSC) of DPNR latex was 0.71% less than that of NR-LA latex due to twice more centrifugation. As a result, the alkalinity and pH of NR-LA latex were higher than those of DPNR latex. The mechanical stability (MST) of both DPNR and NR-LA latices were almost the same. The addition of surfactant, SDS, gave the surface tension of DPNR latex lower than that of NR-LA latex. The nitrogen contents of DPNR and NR-LA latices were 0.039 and 0.408, respectively (Fig. 2). The average particle size and molecular weight of DPNR latex were lower than those of NR-LA latex due to some digested proteins in DPNR.

Latex compound properties

After maturation for two days at 30°C, the DPNR and NR-LA latex compounds had a chloroform number of about 2–3 and a total solids content of $\sim$ 50%. The surface tension, viscosity, and pH of DPNR latex com-
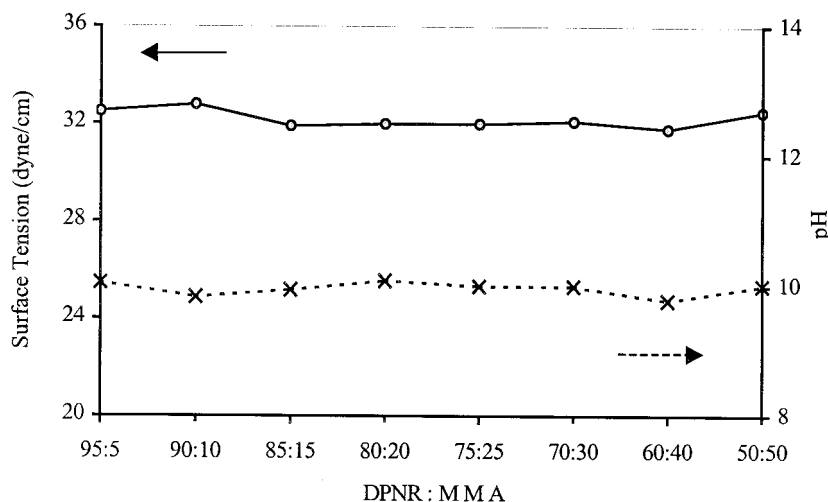
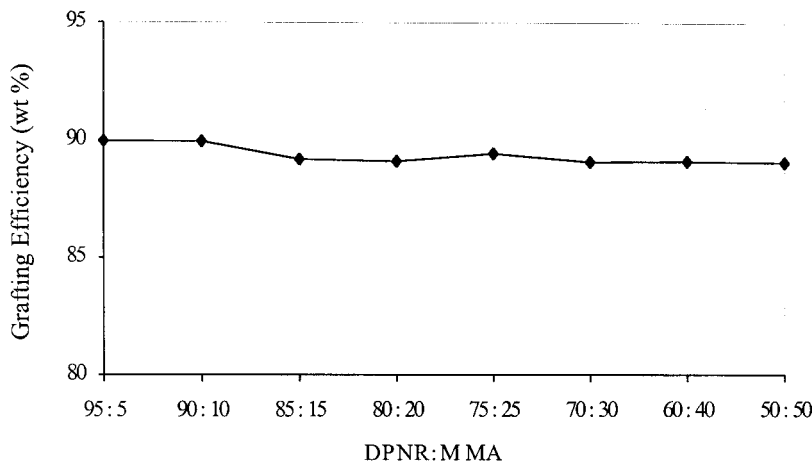**Figure 5** Surface tension and pH of the synthesized graft copolymer latex.

Figure 6 Grafting efficiency of the synthesized graft copolymer latex.

pound (Table II) showed small differences (<10%) from those of NR-LA latex compound.

Poly(methyl methacrylate)

The PMMA obtained from the emulsion polymerization technique had a total solids content of ∼ 40% with a pH of 6.4. The surface tension was about 45 dyn/cm; the viscosity was about 12.5 cP and the molecular weight was 5.37×10^5 g/mol. The average particle size was 0.125 μm, as shown in Figure 3.

Poly(methyl methacrylate)-grafted deproteinized natural rubber

The graft copolymer latices of different weight fractions (DPNR : MMA = 95 : 5, 90 : 10, 85 : 15, 80 : 20, 75 : 25, 70 : 30, 60 : 40, 50 : 50) were synthesized by the emulsion polymerization technique. The synthesized latex had good long-term stability with conversions being higher than 95% and the total solids content of

about 40% (Fig. 4). The synthesized graft copolymer latex (DPNR-*g*-PMMA) was basic alkaline with pH of about 10 and surface tension of about 32 dyn/cm (Fig. 5).

The grafting efficiency obtained from this emulsion polymerization grafting process was between 89 and 90 wt % (Fig. 6). Increasing the weight fraction of MMA would decrease the grafting efficiency because of an increase of ungrafted PMMA during the process of polymerization and a decrease of ungrafted NR. The ungrafted NR was <10 wt %, whereas the ungrafted PMMA was under 4 wt % (Fig. 7). An increase of the ungrafted PMMA with an increase of the weight fraction of MMA monomer may be attributed to a competitive homopolymerization reaction to form PMMA during the graft copolymerization process. Therefore, ungrafted PMMA on DPNR backbone chain was also increased as the weight fraction of MMA monomer increased.
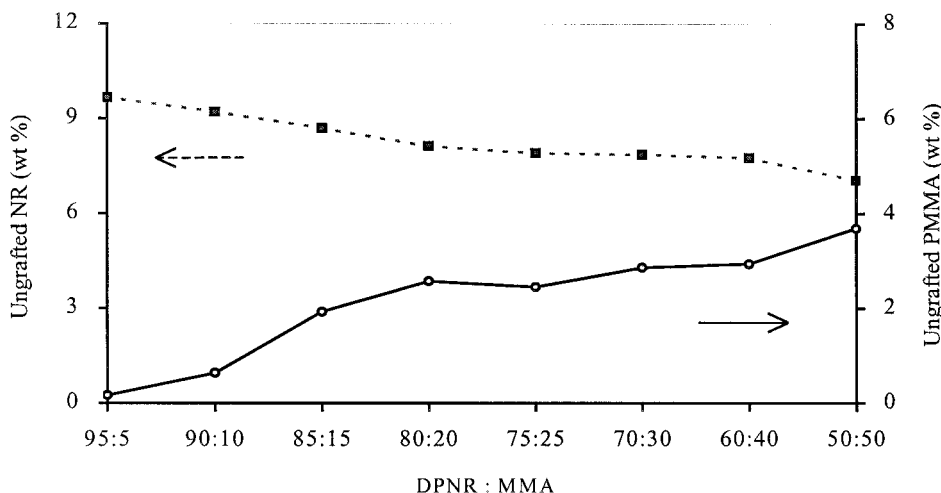

Figure 7 % Ungrafted NR and ungrafted PMMA in the synthesized graft copolymer latex.
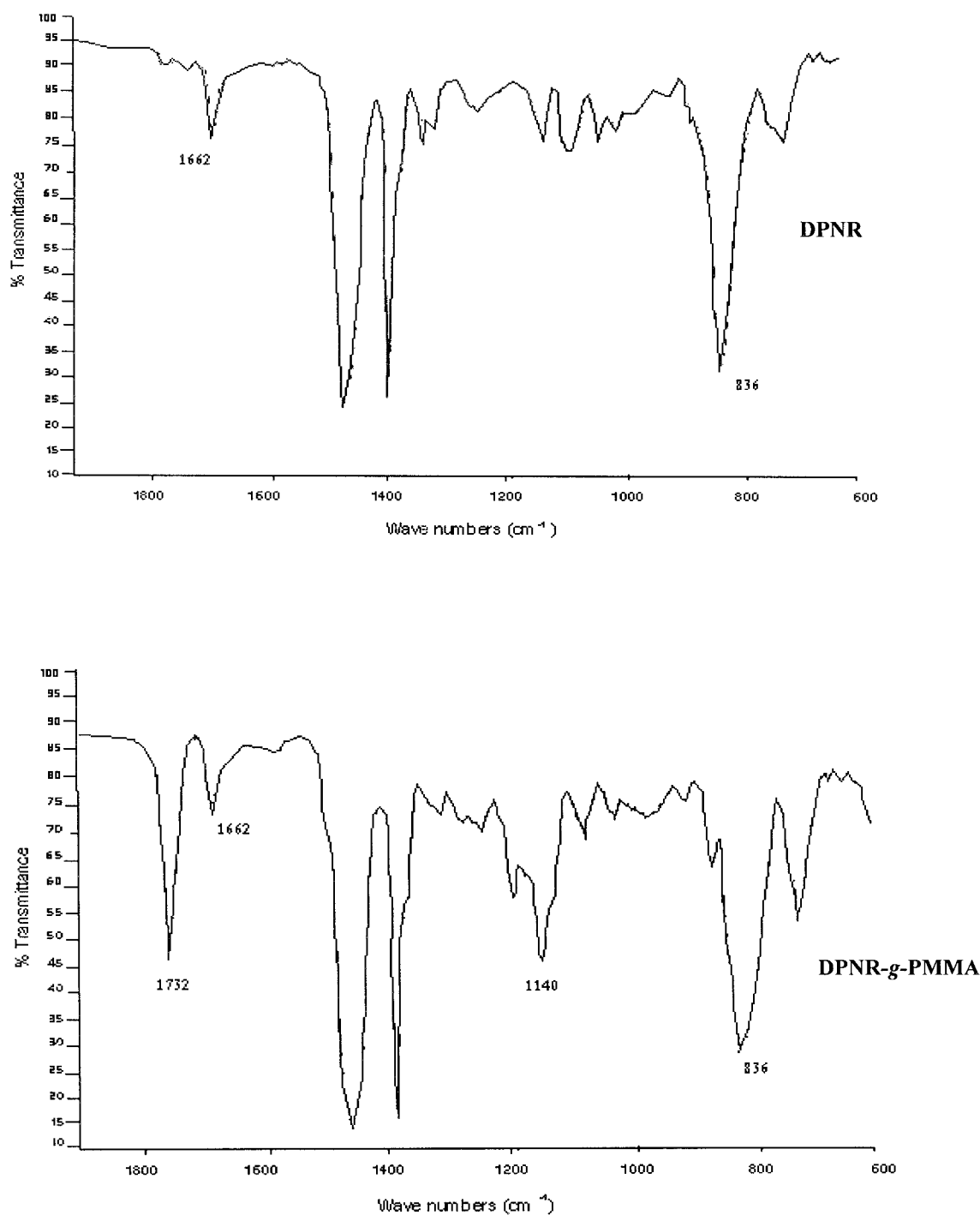
Figure 8   Infrared spectra of DPNR and DPNR-*g*-PMMA.

Characteristics of grafting copolymer (DPNR-*g*PMMA)

The synthesized graft copolymer latex was coagulated by using 10% calcium chloride. After drying, the ungrafted NR and free PMMA homopolymer were removed by means of Soxhlet extraction by using acetone and petroleum ether as solvents, respectively.

The infrared spectra of DPNR-*g*-PMMA showed an absorption peak at 836 cm^{-1} corresponding to C—H bending of NR (Fig. 8). This indicated the presence of *cis*-1,4-polyisoprene in DPNR and DPNR-*g*-PMMA. The intense absorption peaks at 1732 and 1140 cm^{-1} correspond to the carbonyl group (—C=O) and the —C—O functional group of MMA chains in graft copolymer, respectively. The level of grafted PMMA increased with increasing quantity of MMA in the grafting reaction. Hence, when the weight fraction of MMA monomer in the graft copolymer increases, the

TABLE III
Physical Properties of DPNR and NR-LA Films

Properties	DPNR	NR-LA
Before aging		
300% modulus (MPa)	1.56 ± 0.21	1.98 ± 0.258
500% modulus (MPa)	2.23 ± 0.20	3.24 ± 0.22
Tensile strength (MPa)	23.82 ± 0.62	26.35 ± 0.75
Elongation at break (%)	1107 ± 15	1000 ± 30
Friction coefficient	1.46	1.60
After aging at 70°C, 166 h		
300% modulus (MPa)	1.62 ± 0.16	2.69 ± 0.24
500% modulus (MPa)	2.68 ± 0.21	5.57 ± 0.38
Tensile strength (MPa)	18.34 ± 0.25	21.25 ± 0.75
Elongation at break (%)	837 ± 15	720 ± 25
Friction coefficient	1.38	1.49

peak intensity at 1732 cm^{-1} tends to increase, whereas the peak intensity at 836 cm^{-1} tends to decrease.

Appearance of films

The synthesized PMMA latex and DPNR-*g*-PMMA latex with different weight fractions of MMA (DPNR : MMA = 95 : 5, 90 : 10, 85 : 15, 80 : 20, 75 : 25, 70 : 30, 60 : 40, 50 : 50) were cast on plates to observe the film appearance. It was found that the film surface was cracked at high weight fraction of MMA (>15 wt %). Therefore, DPNR and NR-LA blended PMMA latices, and DPNR-*g*-PMMA latex with a maximum weigh fraction of MMA of 15 wt % which would obtain a homogeneous film, were used for coating on DPNR or NR-LA film.

Physical properties of rubber films

DPNR and NR-LA films were prepared to measure the physical properties of rubber. The measurement

procedures employed were according to ASTM D412-98a and ASTM D 573-99. The physical properties are shown in Table III.

It was found that, for both before and after aging at 70°C for 166 h, 500% modulus, tensile strength, and friction coefficient of DPNR films were lower than those of NR-LA films, whereas elongation at break of DPNR film was slightly higher than that of NR-LA film. These results are influenced from some nonrubber (i.e., protein) contents that had been taken away during the process of deproteinization. Some proteins were removed by centrifugation.

The vulcanized rubber films with different weight fractions of PMMA blended or grafted copolymer coated of DPNR and NR-LA were prepared by dipping process. The average results of the stress–strain and friction coefficient properties of vulcanized rubber films are as follows.

Stress–strain properties

The stress–strain properties of the film depend upon the fraction of two components, rubber and PMMA. PMMA is a rigid component that gives a high modulus. Therefore, increasing the weight fraction of PMMA blended into DPNR or NR-LA would increase the modulus of the rubber films (Fig. 9). This is in agreement with Neilsen,[9] who indicated that the greater the modulus ratio, the greater the modulus of the composite. By increasing the PMMA fraction in DPNR-*g*-PMMA used for coating on DPNR or NR-LA film surface, the modulus of the film was also increased. The increasing rate of modulus of PMMA blended DPNR or NR-LA films was higher than that of DPNR-*g*-PMMA coated film because of the fact that PMMA is much more rigid than the rubber grafted
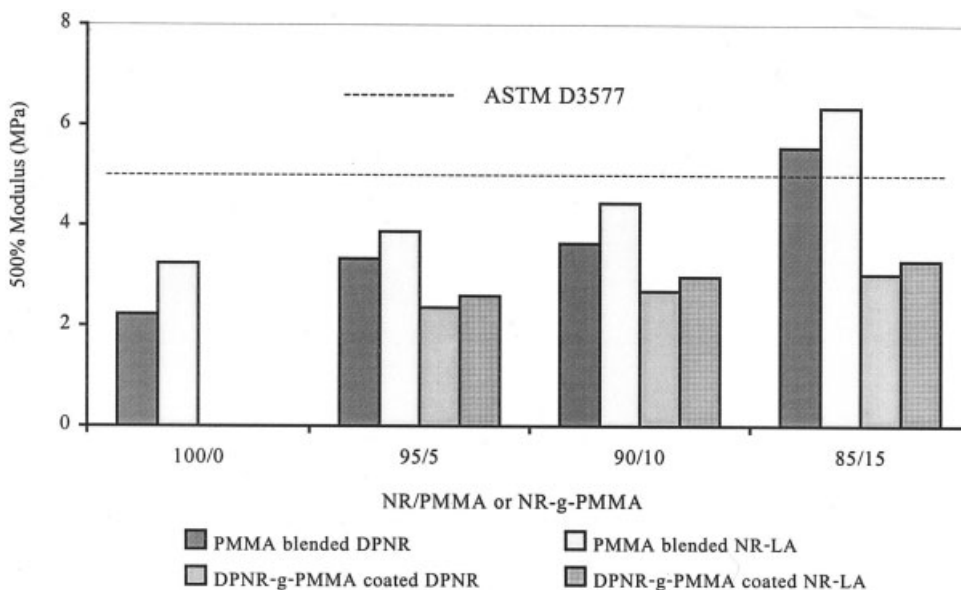
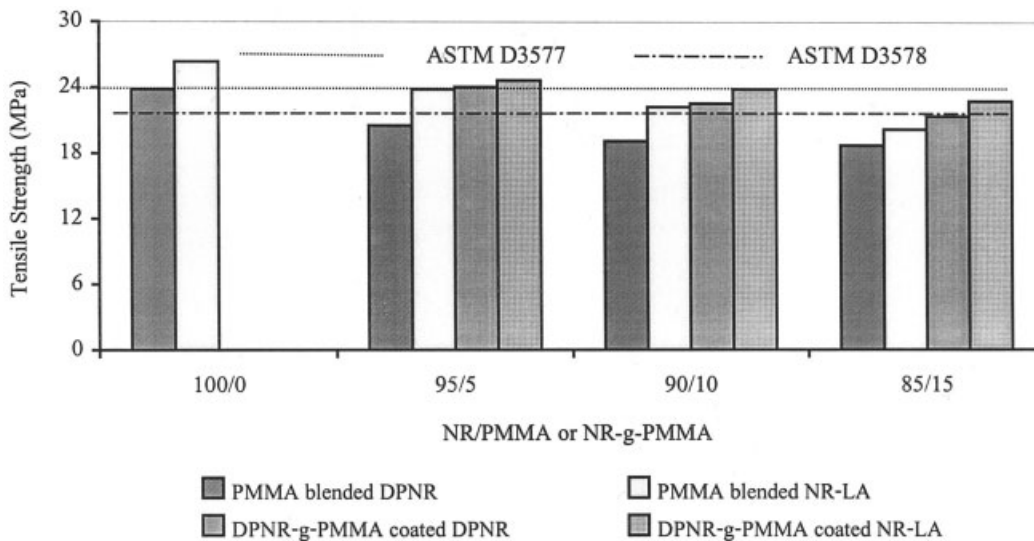**Figure 9** 500% Modulus of rubber films (before aging).

Figure 10 Tensile strength of rubber films (before aging).

copolymer. The higher weight fraction of PMMA in blended latex would give a higher modulus. The films prepared by using 15% weight fraction of PMMA latex blended DPNR or NR-LA compound failed to meet ASTM-3577 standard for rubber surgical gloves. However, DPNR-*g*-PMMA coated DPNR and NR-LA films at all weight fractions could achieve ASTM-3577 standard for rubber surgical gloves.

Modulus of 500% of DPNR-*g*-PMMA coated on DPNR or NR-LA films gradually increased with an increase of the weight fraction of PMMA in the graft copolymer, DPNR-*g*-PMMA (Fig. 9). The tensile strength of the film slightly decreased as PMMA in the graft copolymer increased (Fig. 10). Because PMMA is

the hard segment, the bond strength of the film would subsequently decrease while increasing the extension of the rubber film. In addition, the elongation at break of DPNR-*g*-PMMA coated DPNR or NR-LA films slowly decreased with an increase of PMMA in the graft copolymer (Fig. 11).

As some proteins on the rubber particles of DPNR were removed by enzyme, the tensile strength of DPNR film was lower than that of NR-LA film (Fig. 10). Increasing the weight fraction of PMMA blended DPNR or NR-LA would decrease the tensile strength of the rubber film. Also an increase of PMMA on the DPNR-*g*-PMMA coated on the DPNR or NR-LA film would decrease the tensile strength of the DPNR or
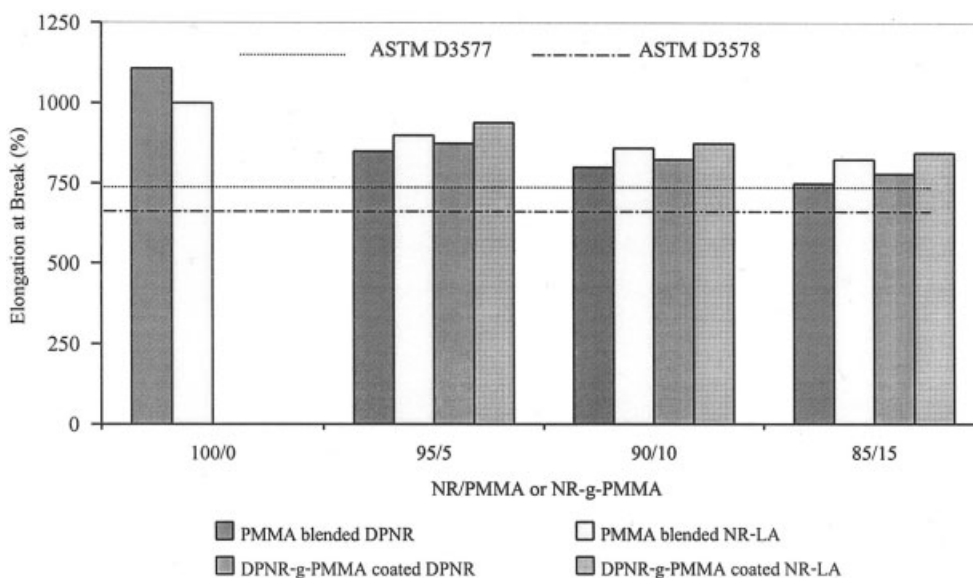

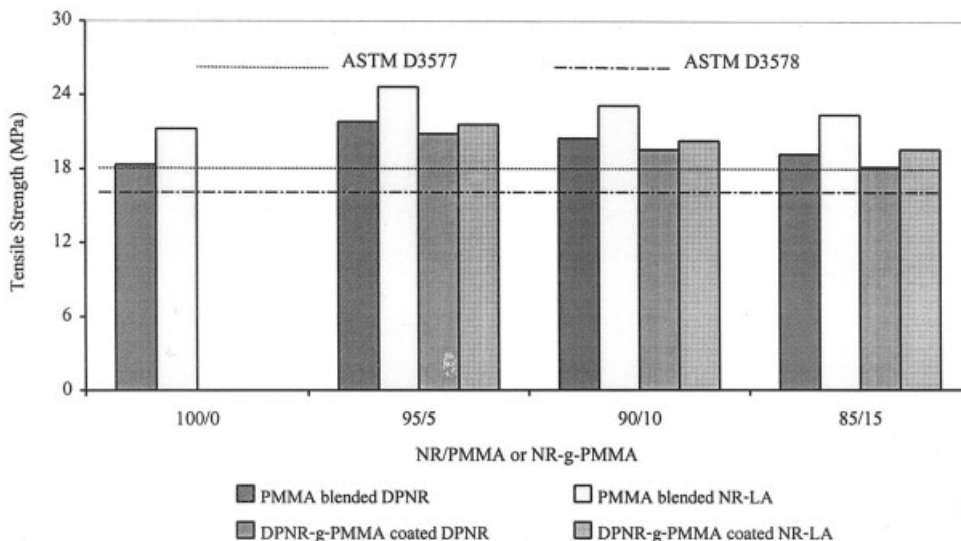**Figure 11** Elongation at break of rubber films (before aging).

Figure 12 Tensile strength of rubber films (after aging).

NR-LA film. This could be a result of increasing the hard rigid PMMA polymer fraction that caused a decrease in the compatibility of rubber and, hence, a reduction in the strength of rubber. In the case of PMMA blended DPNR or NR-LA latex, the hard rigid PMMA polymer was possibly only located between the rubber particle surface without any significant interaction with the rubber molecules. The strength properties were therefore lower than those of graft copolymer. After aging at 70°C for 166 h, the tensile strength of both PMMA blended DPNR or NR-LA and DPNR-*g*-PMMA coated on DPNR or NR-LA films were decreased (Fig. 12). The graft copolymer, DPNR-*g*-PMMA, appeared as only a thin film coating on the

DPNR or NR-LA films. Therefore, it could not improve the strength of the rubber film. However, all the tensile strength properties meet ASTM-3577 and ASTM-3578 standards for rubber surgical gloves and examination gloves.

The elongation at break of DPNR film (Fig. 11) was higher than that of NR-LA film due to its lower crosslink density.[10] The hard rigid PMMA would decrease elongation of rubber. Thus, an increase in weight fraction of PMMA blended into DPNR or NR-LA latex compound would slightly decrease elongation at break of the film. The elongation at break of DPNR-*g*-PMMA coated on DPNR film was lower than that of DPNR-*g*-PMMA coated on NR-LA film because
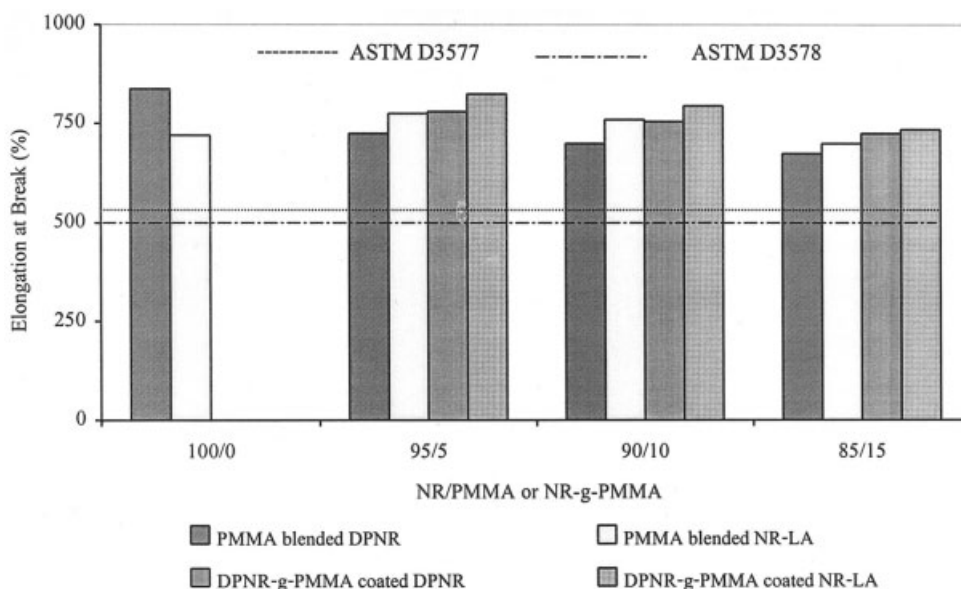

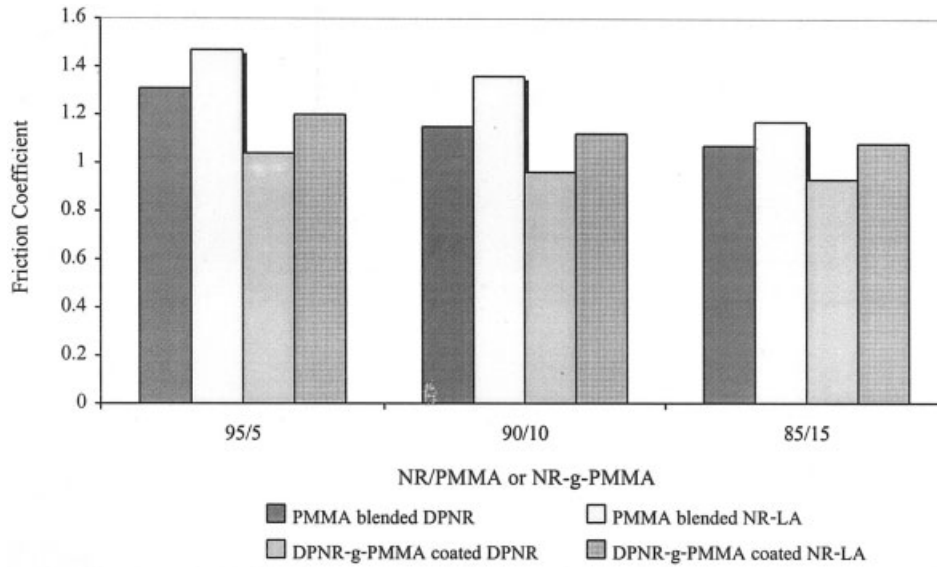**Figure 13** Elongation at break of rubber films (after aging).

Figure 14 Friction coefficient of rubber films (before aging).

a decrease in nitrogen content in DPNR would also decrease the adherence between the film surfaces. After aging at 70°C for 166 h, the elongation at break of PMMA blended DPNR or NR-LA remained lower than that of DPNR or NR-LA coated with DPNR-*g*-PMMA (Fig. 13) because PMMA is a thermoplastic polymer that cannot be extended as good as NR graft copolymer.

Friction coefficient of rubber

The friction of rubber depends on the viscous properties of the rubber compound.[11] DPNR contains lower protein than NR-LA. Therefore, it will be adhered less to the DPNR rubber surface and gives the friction coefficient of DPNR lower than that of NR-LA (Fig. 14). Blended PMMA into DPNR or NR-LA would slightly decrease the friction coefficient of the rubber due to PMMA, a hard rigid thermoplastic polymer giving less friction coefficient than rubbery materials.[12] The friction coefficient of DPNR-*g*-PMMA coated onto DPNR or NR-LA film surface was found to be smaller than that of PMMA blended into DPNR or NR-LA. After aging at 70°C for 166 h, the friction coefficient of those rubber films showed no significant differences (Fig. 15).
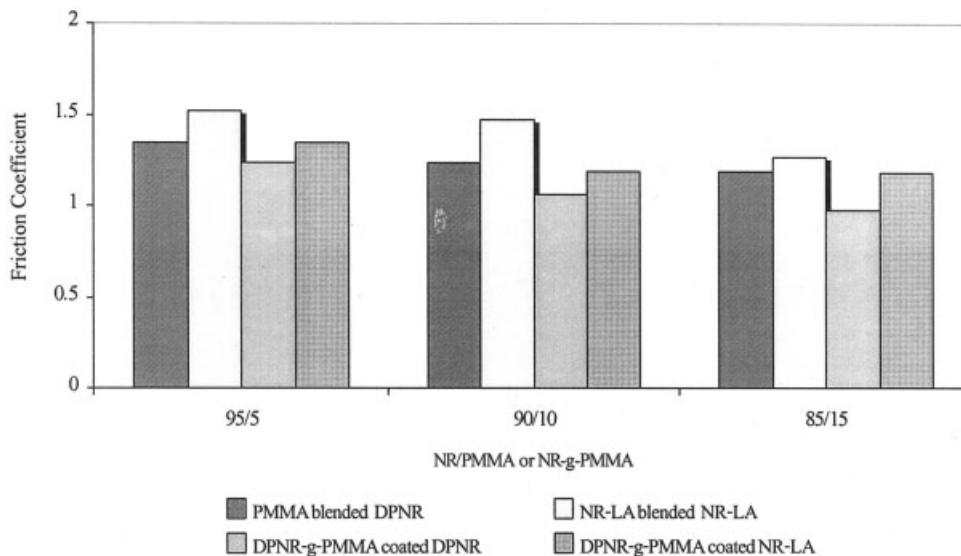

Figure 15 Friction coefficient of rubber films (after aging).

TABLE IV
The Physical Properties of Rubber Films Pass/Fail ASTM D3577-78a Specification

Samples	500% Modulus (before aging)				T.S. (before, after aging)				%E.B. (before, after aging)			
	100/0	95/5	90/10	85/15	100/0	95/5	90/10	85/15	100/0	95/5	90/10	85/15
DPNR blended PMMA	✓	✓	✓	X	X,✓	X,✓	X,✓	X,✓	✓,✓	✓,✓	✓,✓	✓,✓
NR-LA blended PMMA	✓	✓	✓	X	✓,✓	X,✓	X,✓	X,✓	✓,✓	✓,✓	✓,✓	
DPNR coated DPNR-*g*-PMMA	✓	✓	✓	✓	X,✓	✓,✓	X,✓	X,✓	✓,✓	✓,✓	✓,✓	✓,✓
NR-LA coated DPNR-*g*-PMMA	✓	✓	✓	✓	✓,✓	✓,✓	X,✓	X,✓	✓,✓	✓,✓	✓,✓	✓,✓

Note. ✓, pass; X, fail.

ASTM standard specification of rubber glove

The specification required for surgical gloves and examination gloves are ASTM D3577-78a and ASTM D3578-99 standards. The physical properties of surgical gloves required by ASTM D3577-78a is limited to a maximum value of 500% modulus at 5.5 MPa, whereas examination glove is not assigned. The tensile strength and elongation at break of the surgical glove are limited to a minimum at 24 MPa and 750%, respectively, whereas those of the examination glove are limited to a minimum at 21 MPa and 700%, respectively. After aging at 70°C for 166 h, the tensile strength and elongation at break are limited to a minimum at 18 MPa and 560% for ASTM D3577-78a, and at 16 MPa and 500% for ASTM D3578 99, respectively.

From this work, it was found that the rubber films with the physical properties achieved the ASTM D3577-78a specification (as shown in Table IV) were the rubber film prepared from NR-LA compound and the rubber film prepared from DPNR-*g*-PMMA (weight fraction of 95/5) coated on DPNR film, also the rubber film that was prepared from DPNR-*g*-PMMA (weight fraction of 95/5) coated on NR-LA film.

The rubber films with the physical properties, which achieved the ASTM D3578-99 specification (shown in Table V), were as follows: the rubber film prepared from NR-LA compound; the rubber film prepared from DPNR compound; the rubber film prepared from blended PMMA/NR-LA (weight fraction of 90/10); and the rubber film prepared from DPNR-*g*-PMMA (weight fraction of 85/15) coated on DPNR or NR-LA film.

For all the weight fractions of PMMA blended into DPNR or NR-LA compound, the film properties could not achieve the ASTM D3577-78a specification for the surgical glove.

CONCLUSION

A homogenous film obtained from blending PMMA into NR-LA latex compound could achieve the rubber examination glove specification at the fraction of about 90 : 10 (w/w). DPNR-*g*-PMMA coated on DPNR and NR-LA films could also achieve rubber surgical glove specification at a weight fraction of 95/5 and examination glove specification at a maximum weight fraction of 85/15. The friction coefficient

TABLE V
The Physical Properties of Rubber Films Pass/Fail ASTM D3578-99 Specification

Samples	500% Modulus (before aging)				T.S. (before, after aging)				%E.B. (before, after aging)			
	100/0	95/5	90/10	85/15	100/0	95/5	90/10	85/15	100/0	95/5	90/10	85/15
DPNR blended PMMA	✓	✓	✓	X	✓,✓	X,✓	X,✓	X,✓	✓,✓	✓,✓	✓,✓	✓,✓
NR-LA blended PMMA	✓	✓	✓	X	✓,✓	✓,✓	✓,✓	X,✓	✓,✓	✓,✓	✓,✓	✓,✓
DPNR coated DPNR-*g*-PMMA	✓	✓	✓	✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓
NR-LA coated DPNR-*g*-PMMA	✓	✓	✓	✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓	✓,✓

Note. ✓, pass; X, fail.

of DPNR film was lower than that of NR-LA film. By coating DPNR-*g*-PMMA onto DPNR and NR-LA films, the friction coefficient could be decreased more than by blending PMMA into DPNR and NR-LA.

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