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APPENDICES

APPENDIX A

Overall composition of rubbers

Table A-1 Properties of natural rubber latex concentrated

Properties	Test results
Total solids content, %	61.88
Dry rubber content, %	60.14
Non rubber solids, %	1.824
Ammonia content (on total weight), %	0.669
Ammonia content (on water phase), %	1.756
pH value	10.39
KOH number	0.679
Volatile fatty acid number (VFA)	0.027
Mechanical stability at 55% TS. Sec	830
Specific gravity at 25°C	0.943
Viscosity (60% TS, spindle no.1.60 rpm),cps	78.50
Coagulum content (80 mesh), ppm	27.00

All tests are performed according to relevant ISO 2004-1997(E) specification.

APPENDIX B

Design of glass reactor

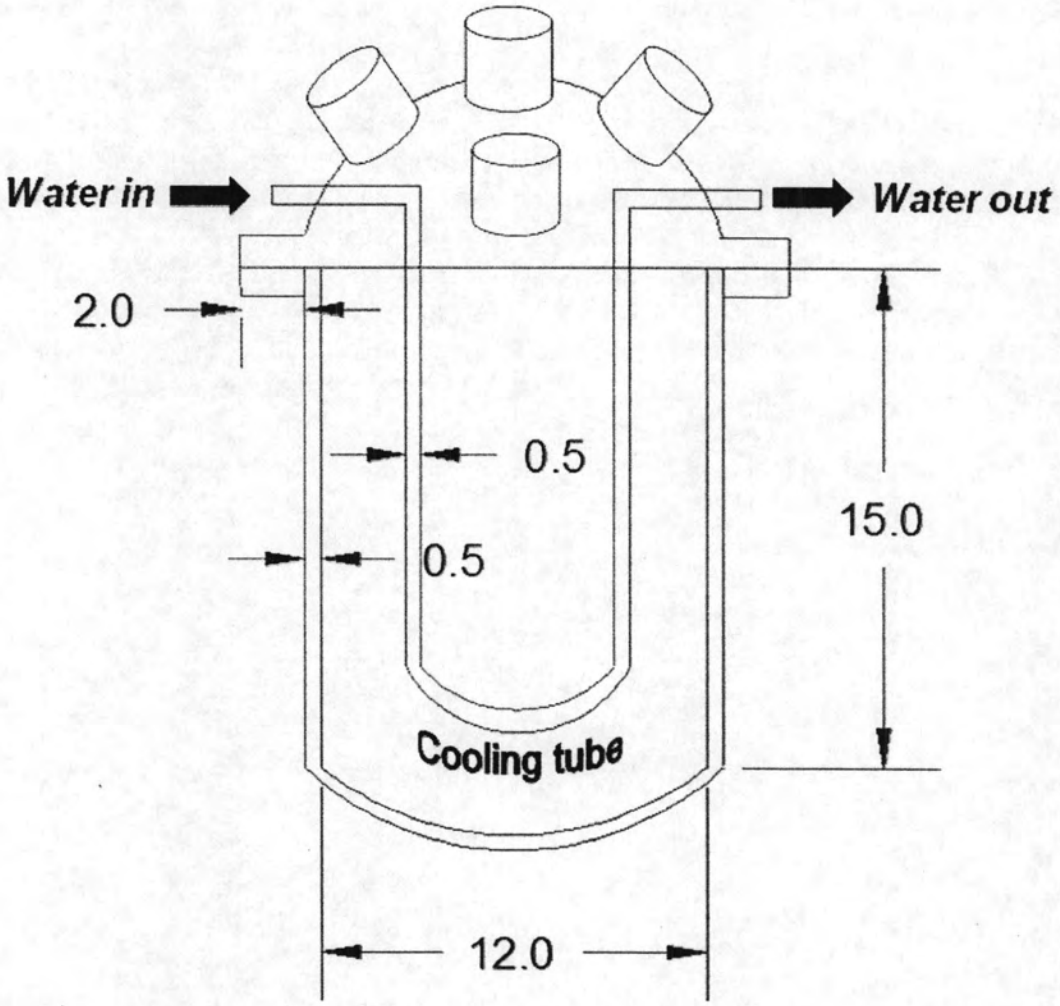


Figure B-1 Size of glass reactor (cm).

APPENDIX C

Mechanism of graft copolymerization

The graft copolymerization of methyl methacrylate (MMA) onto natural rubber (NR) was prepared by seed emulsion polymerization. The grafting requires an extractable hydrogen, such as present on the backbone of polyisoprene, polybutadiene, and polystyrene. The objective is to create a graft site at the particle-water interface, so that efficient subsequent propagation from this graft site with a hydrophilic monomer is then possible. In addition, a transfer reaction leading to homopolymer in the water phase, and cross-linking of the seed polymer, may also occur. This is shown schematically in Figure C-1.

To control the locus of radical formation, a redox initiation system was selected consisting of a hydrophilic reducing agent and a hydrophobic oxidizing agent. It was proposed that a couple such as cumene hydroperoxide (CHPO) and tetraethylenepentamine (TEPA), hydrophobic and hydrophilic species, respectively, will generate radicals mainly at the polymer/water interface due to the partitioning of CHPO and TEPA primarily into the organic phase and aqueous phases, respectively. The grafting process can occur either through abstraction or addition, as shown in Figure C-2. Bulky initiator radicals, such as the cumyloxy radical from the present system, favor abstraction compared to addition. The primary cumyloxy radical will readily abstract protons from the polymer backbone, creating a grafting site accessible to aqueous monomer. Further addition of hydrophilic monomer will extend the grafted chain into the aqueous phase. A second criterion for effective grafting is that the monomer to be used for the second stage graft be sufficiently reactive to propagate readily with the radical resulting from the abstraction step. In the present case, that radical is allylic (Figure C-2), and hence it is essential to choose the second stage monomer to be sufficiently reactive: the stability of the allylic radical may mean that it could have a very slow reinitiation rate coefficient with some monomers. Thus monomers such as vinyl esters, where the radical is reactive but the monomer is not, would be inappropriate, while acrylates and, to a lesser extent, methacrylates are more reactive monomers (Lamb et al., 2001).

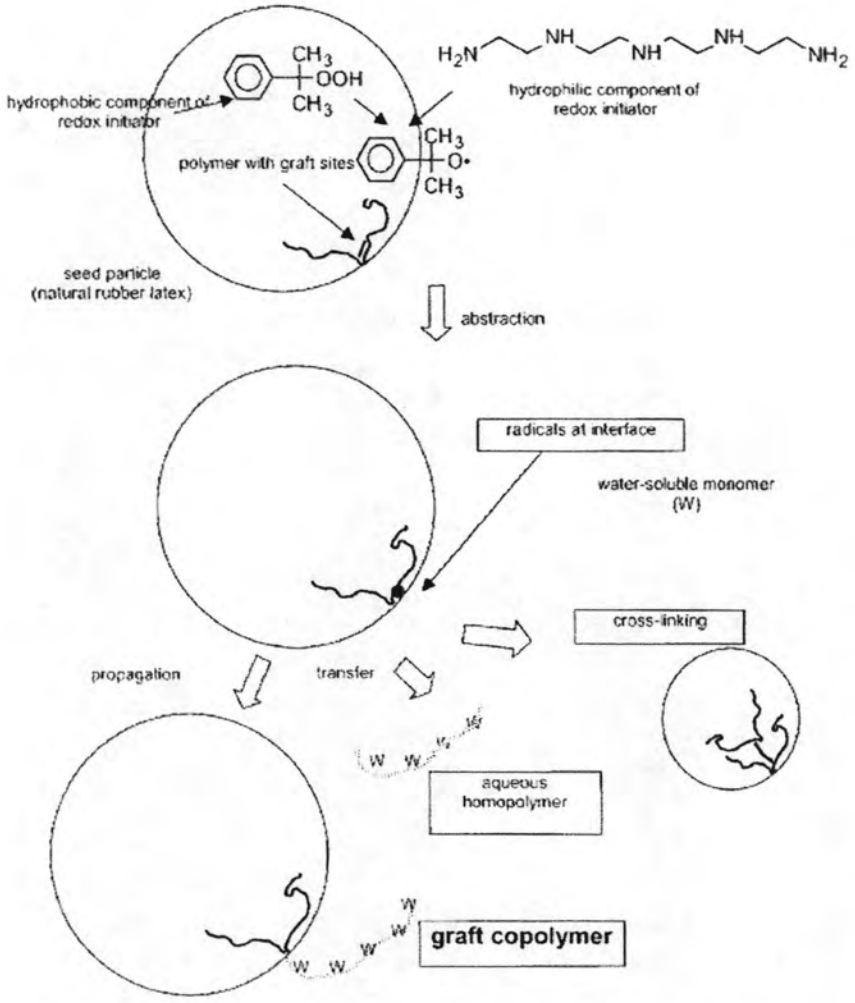


Figure C-1 Schematic of seed emulsion polymerization leading to graft copolymer. Water-phase polymerization to form homopolymer of the water-soluble monomer, W, and cross-linking of the graftable seed polymer within the particle phase are also possible (Lamb et al., 2001).

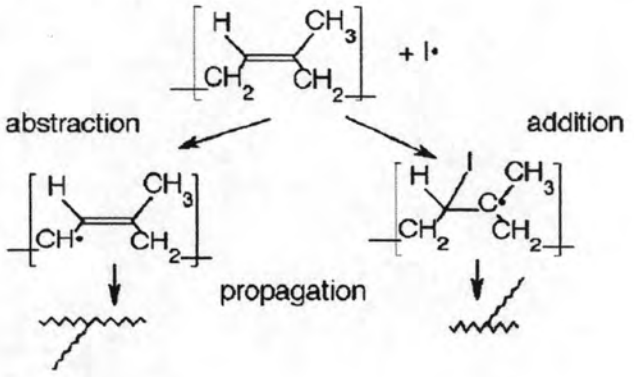
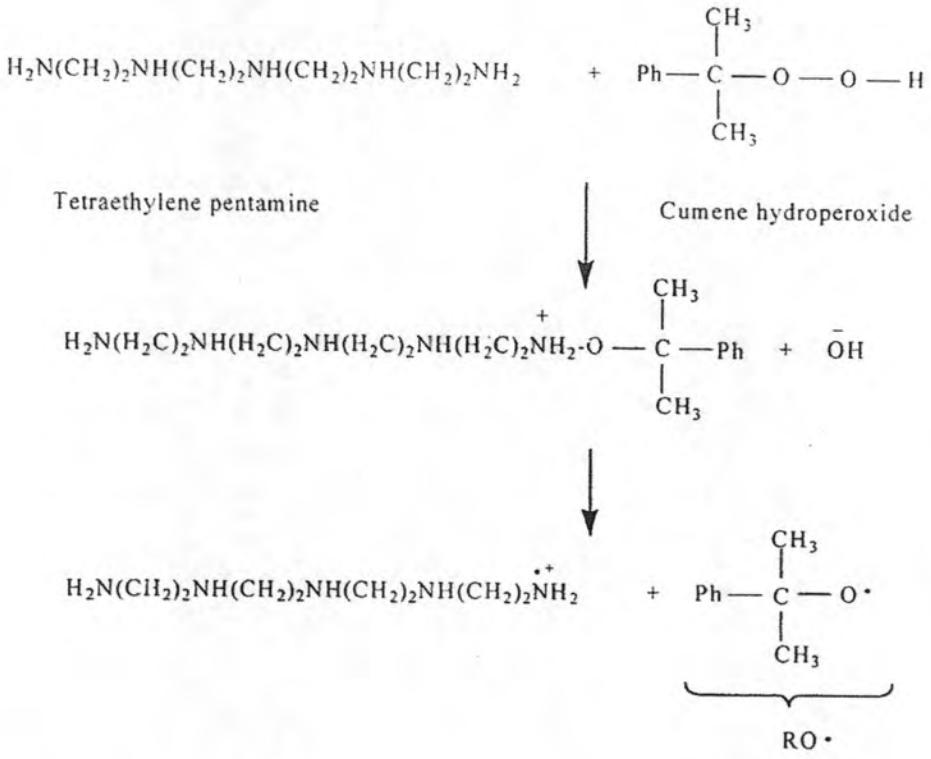


Figure C-2 Abstraction and addition reactions which can lead to grafting in systems with a labile hydrogen such as polyisoprene (Lamb et al., 2001).

The following reaction scheme is proposed for the graft copolymerization of MMA onto NR by the free radical polymerization (Kanchana Eawsuwan, 2003):

a) Initiation

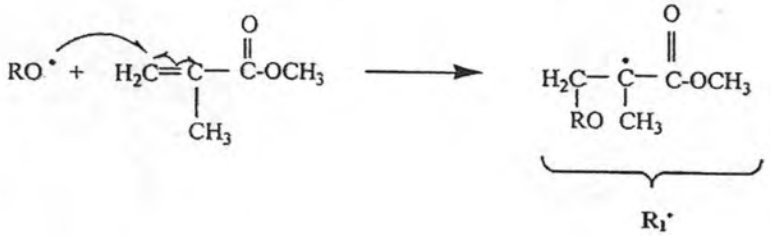
Initiating radicals are then produced by the resulting redox catalyst system, i.e. by one electron oxidation-reduction reactions. The CHPO in the dilute aqueous solution induced by TEPA decompose to yield alkoxy radicals ($RO^{\bullet}$) as shown in Eq.1-2. The alkoxy radical might attack MMA monomer to form MMA radical , $R_1^{\bullet}$ (Eq. 3) or attack NR molecule (Eq. 4) leading to the formation of the active grafting site of NR macroradical ($R_2^{\bullet}$) for graft copolymerization.



(1)

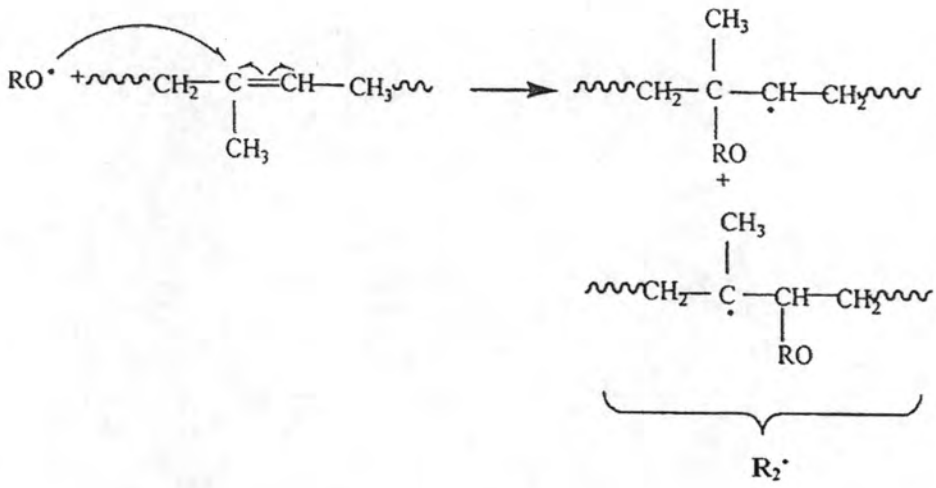
(2)

i) Attacking monomer



(3)

ii) Attacking rubber



(4)

b) Propagation

Both free and grafted chains are in the same environment and are presumed to grow at equivalent rates as described by:

i) Propagation of homopolymerization

Polymerization of monomer itself, poly(methyl methacrylate) (PMMA) can be occurred. It is proposed that $R_1^{\bullet}$ attacks double bond of the MMA to form the PMMA radical which then might attack other MMA monomers (Eq.5).

ii) Propagation of graft copolymerization

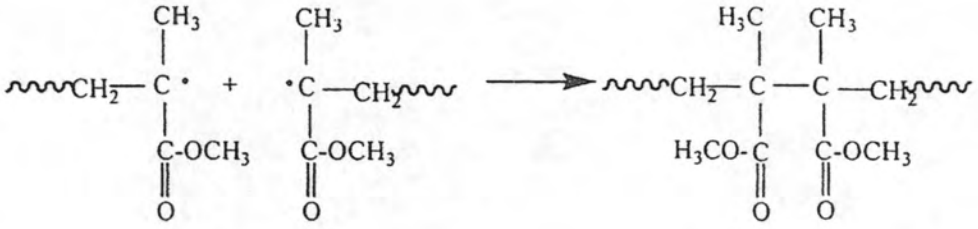
The graft copolymerization can be occurred when $R_2^{\bullet}$ attacks double bond of the MMA to form the graft copolymer radical (Eq.6) and then might attack another MMA to form the longer grafted chain.

(5)

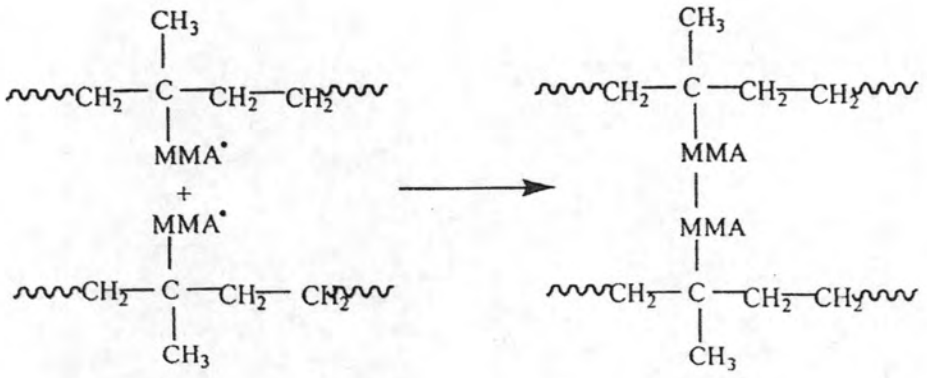
(6)

c) Termination

A termination reaction always involves two radicals reacting by recombination. In general case, a bond is formed between two growing radicals which might occur the recombination of MMA macroradicals (Eq.7) or graft copolymer radicals (cross-linking) as shown in Eq.8 leading to the termination of homopolymerization and graft copolymerization, respectively.



(7)



(8)

APPENDIX D

Data of graft copolymerization

Table D-1 Data for dielectric properties of chemicals for graft copolymerization

Chemicals	Replicate	Resonant frequency (MHz)	Quality factor	Dielectric constant (ε_r')	Loss factor (ε_r'')	Loss tangent ($\tan\delta = \varepsilon_r'/\varepsilon_r''$)
SDS	1	2478.32	98.46	1.4273	0.0357	0.0250
	2	2476.53	95.42	1.4452	0.0428	0.0296
	3	2478.52	96.98	1.4122	0.0393	0.0278
	4	2481.03	99.90	1.3699	0.0330	0.0241
	5	2479.94	98.14	1.3882	0.0368	0.0265
	Ave.	**2478.87**	**97.78**	**1.4085**	**0.0375**	**0.0266**
KOH	1	2407.99	92.04	2.6531	0.0326	0.0123
	2	2438.16	95.55	2.1071	0.0335	0.0159
	3	2431.26	98.09	2.2245	0.0251	0.0113
	4	2424.52	99.61	2.3431	0.0193	0.0082
	5	2419.90	95.89	2.4267	0.0268	0.0110
	Ave.	**2424.37**	**96.24**	**2.3509**	**0.0274**	**0.0117**
NR latex	1	2270.65	6.81	2.4788	3.2995	1.3311
	2	2290.02	8.30	2.7559	2.7143	0.9849
	3	2290.86	8.60	2.8622	2.6256	0.9173
	4	2296.13	8.77	2.8197	2.5632	0.9091
	5	2300.38	9.52	2.9264	2.3568	0.8054
	Ave.	**2289.61**	**8.40**	**2.7686**	**2.7119**	**0.9895**
i-propanol	1	2470.03	39.45	1.5412	0.3768	0.2445
	2	2488.10	64.37	1.2555	0.1560	0.1242
	3	2485.61	70.51	1.2679	0.1261	0.0994
	4	2488.60	65.94	1.2458	0.1478	0.1186
	5	2477.91	39.41	1.2854	0.3740	0.2910
	Ave.	**2482.05**	**55.94**	**1.3192**	**0.2361**	**0.1755**
DI water	1	1858.46	9.26	37.0533	19.9722	0.5390
	2	1845.46	8.52	33.1261	19.7068	0.5949
	3	1852.80	10.86	38.6196	16.8137	0.4354
	4	1849.16	10.90	39.4016	17.3090	0.4393
	5	1840.49	10.90	40.9313	18.5038	0.4521
	Ave.	**1849.27**	**10.09**	**37.8263**	**18.4611**	**0.4921**

Table D-1 Data for dielectric properties of chemicals for graft copolymerization (continued)

Chemicals	Replicate	Resonant frequency (MHz)	Quality factor	Dielectric constant (ε_r')	Loss factor (ε_r'')	Loss tangent (tan $\delta = \varepsilon_r'/\varepsilon_r''$)
CHPO	1	2311.02	6.53	1.6124	3.2933	2.0425
	2	2308.94	6.40	1.5887	3.3592	2.1144
	3	2307.79	6.28	1.5467	3.4182	2.2101
	4	2311.99	6.82	1.7418	3.1687	1.8192
	5	2310.11	6.78	1.7612	3.1909	1.8118
	Ave.	**2309.97**	**6.56**	**1.6502**	**3.2861**	**1.9996**
TEPA	1	2292.87	11.96	5.4546	2.1152	0.3878
	2	2291.80	12.53	5.4064	1.9969	0.3694
	3	2380.97	11.33	4.0792	2.0727	0.5081
	4	2391.29	9.99	3.8561	2.3583	0.6116
	5	2369.03	10.43	4.3993	2.3331	0.5303
	Ave.	**2345.19**	**11.25**	**4.6391**	**2.7524**	**0.4814**

Table D-2 Data for graft copolymerization of MMA onto NR obtained from conventional method and microwave method at 60°C

Exp.	NR (g)	%DRC	NR content (g)	MMA (g)	Graft product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
Conventional method																
GNR_C10	50.95	60.74	30.95	15.23	42.43	75.39	0.5294	0.2804	0.2096	39.59	47.03	13.37	11.48	5.67	5.81	50.59
	50.13	60.74	30.45	15.35	41.64	72.91	0.5123	0.2598	0.1956	38.18	49.29	12.53	11.19	5.22	5.97	53.38
GNR_C20	50.89	60.74	30.91	15.06	42.41	76.36	0.5425	0.3004	0.2306	42.51	44.63	12.87	11.50	5.46	6.04	52.55
	50.13	60.74	30.45	15.02	40.86	69.31	0.5515	0.3125	0.2523	45.75	43.34	10.92	10.41	4.46	5.95	57.17
GNR_C30	50.67	60.74	30.78	15.04	42.61	78.67	0.5141	0.3071	0.2472	48.08	40.26	11.65	11.83	4.96	6.87	58.05
	50.12	60.74	30.45	15.10	41.68	74.38	0.5168	0.2987	0.2378	46.01	42.20	11.78	11.23	4.91	6.32	56.28
GNR_C40	50.08	60.74	30.42	15.00	41.03	70.79	1.0695	0.7548	0.6468	60.48	29.42	10.10	10.61	4.14	6.47	60.96
	50.15	60.74	30.46	15.14	40.58	66.82	1.1021	0.7328	0.6274	56.93	33.51	9.56	10.12	3.88	6.24	61.65
GNR_C60	50.44	60.74	30.64	15.09	41.82	74.10	0.5335	0.3788	0.3285	61.57	29.00	9.43	11.18	3.94	7.24	64.75
	50.24	60.74	30.51	15.14	40.96	68.99	0.5478	0.3654	0.3125	57.05	33.30	9.66	10.45	3.96	6.49	62.14
GNR_C7h	50.50	60.74	30.67	15.06	42.58	79.10	0.5503	0.4227	0.3761	68.34	23.19	8.47	11.91	3.61	8.30	69.72
	50.65	60.74	30.77	15.10	41.85	73.39	0.5401	0.4058	0.3584	66.36	24.87	8.78	11.08	3.67	7.41	66.86

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-2 Data for graft copolymerization of MMA onto NR obtained from conventional method and microwave method at 60°C (continued)

Exp.	NR (g)	%DRC	NR content (g)	MMA (g)	Graft product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
Microwave method																
GNR_M05	50.42	60.74	30.62	15.10	40.52	65.54	0.5264	0.2831	0.2005	38.09	46.22	15.69	9.90	6.36	3.54	35.75
	50.57	60.74	30.72	15.02	39.56	58.88	0.5215	0.2756	0.1996	38.27	47.15	14.57	8.84	5.77	3.08	34.82
GNR_M10	50.82	60.74	30.87	15.05	41.25	69.01	0.5108	0.3146	0.2560	50.12	38.41	11.47	10.38	4.73	5.65	54.43
	50.13	60.74	30.45	15.15	40.56	66.77	0.5325	0.3356	0.2686	50.44	36.98	12.58	10.11	5.10	5.01	49.54
GNR_M20	50.54	60.74	30.70	15.01	42.74	80.21	0.5085	0.3971	0.3390	66.67	21.91	11.43	12.04	4.88	7.16	59.44
	50.24	60.74	30.52	15.12	41.89	75.22	0.5105	0.3854	0.3257	63.80	24.51	11.69	11.37	4.90	6.47	56.93
GNR_M30	50.08	60.74	30.42	15.02	44.09	91.01	0.4983	0.3889	0.3210	64.42	21.95	13.63	13.67	6.01	7.66	56.05
	50.24	60.74	30.51	15.37	43.86	86.84	0.5148	0.3902	0.3187	61.91	24.20	13.89	13.35	6.09	7.25	54.36
GNR_M40	50.31	60.74	30.56	15.01	40.93	69.08	0.5326	0.3772	0.3154	59.22	29.18	11.60	10.37	4.75	5.62	54.20
	50.46	60.74	30.65	15.29	41.15	68.69	0.5263	0.3744	0.3105	59.00	28.86	12.14	10.50	5.00	5.51	52.42
GNR_M60	50.40	60.74	30.61	15.06	41.02	69.13	0.5543	0.3753	0.3060	55.20	32.29	12.50	10.41	5.13	5.28	50.73
	50.26	60.74	30.53	15.13	40.64	66.84	0.5214	0.3423	0.2745	52.65	34.35	13.00	10.11	5.28	4.83	47.75

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-3 The average of %GNR, %free NR, %free PMMA, %GE and %conversion obtained from conventional method and microwave method

Exp.	Ave. %GNR	Ave. %Free NR	Ave. %Free PMMA	Ave. %GE	Ave. %Conversion
Conventional method					
GNR_C10	38.89	48.16	12.95	51.98	74.15
GNR_C20	44.13	43.98	11.89	54.86	72.83
GNR_C30	47.05	41.23	11.72	57.16	76.52
GNR_C40	58.70	31.47	9.83	61.31	68.80
GNR_C60	59.31	31.15	9.54	63.44	71.54
GNR_C7h	67.35	24.03	8.62	68.29	76.25
Microwave method					
GNR_M05	38.18	46.69	15.13	35.28	62.21
GNR_M10	50.28	37.69	12.03	51.99	67.89
GNR_M20	65.23	23.21	11.56	58.19	77.72
GNR_M30	63.16	23.08	13.76	55.20	88.92
GNR_M40	59.11	29.02	11.87	53.31	68.89
GNR_M60	53.93	33.32	12.75	49.24	67.98

Table D-4 2^4 factorial design data for graft copolymerization of MMA onto NR induced by microwave irradiation at 60°C

Exp.	NR (g)	%DRC	NR content (g)	MMA (g)	Graft product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
GNR 01	50.73	60.74	30.81	7.59	33.69	37.96	0.5644	0.2370	0.2071	36.69	58.01	5.30	2.88	1.78	1.09	38.01
	50.29	60.74	30.55	7.50	34.05	46.67	0.5234	0.2296	0.1976	37.75	56.13	6.11	3.50	2.08	1.42	40.55
GNR 02	50.65	60.74	30.77	7.51	35.80	67.00	0.4996	0.2259	0.1661	33.25	54.78	11.97	5.03	4.29	0.75	14.86
	50.33	60.74	30.57	7.49	34.93	58.21	0.5021	0.2195	0.1686	33.58	56.28	10.14	4.36	3.54	0.82	18.75
GNR 03	50.99	60.74	30.97	22.50	45.45	64.34	0.5356	0.3796	0.3063	57.19	29.13	13.69	14.48	6.22	8.26	57.04
	50.49	60.74	30.67	22.51	42.77	53.75	0.5286	0.3668	0.2963	56.05	30.61	13.34	12.10	5.70	6.40	52.86
GNR 04	50.96	60.74	30.95	22.50	53.30	99.33	0.5282	0.3393	0.2378	45.02	35.76	19.22	22.35	10.24	12.11	54.17
	49.99	60.74	30.36	22.54	50.95	91.31	0.5302	0.3297	0.2298	43.34	37.82	18.84	20.59	9.60	10.99	53.36
GNR 05	50.85	60.74	30.89	7.53	33.45	34.03	0.5096	0.2358	0.2100	41.21	53.73	5.06	2.56	1.69	0.87	33.92
	50.55	60.74	30.70	7.51	33.29	34.41	0.5106	0.2222	0.1956	38.31	56.48	5.21	2.59	1.73	0.85	32.91
GNR 06	50.96	60.74	30.95	7.50	33.47	33.54	0.5398	0.2459	0.2113	39.14	54.45	6.41	2.52	2.15	0.37	14.72
	50.23	60.74	30.51	7.49	32.85	31.22	0.5408	0.2324	0.2000	36.98	57.03	5.99	2.34	1.97	0.37	15.82
GNR 07	50.54	60.74	30.70	22.51	45.48	65.68	0.5098	0.3902	0.3398	66.65	23.46	9.89	14.78	4.50	10.29	69.58
	49.96	60.74	30.34	22.55	43.83	59.81	0.5110	0.3828	0.3289	64.36	25.09	10.55	13.49	4.62	8.86	65.72
GNR 08	50.31	60.74	30.56	22.51	46.27	69.79	0.5140	0.4496	0.3600	70.04	12.53	17.43	15.71	8.07	7.64	48.66
	50.11	60.74	30.43	22.51	44.65	63.16	0.5095	0.4284	0.3430	67.32	15.92	16.76	14.22	7.48	6.73	47.35

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-4 2^4 factorial design data for graft copolymerization of MMA onto NR induced by microwave irradiation at 60°C (continued)

Exp.	NR (g)	%DRC	NR content (g)	MMA (g)	Graft product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
GNR 09	50.21	60.74	30.49	7.51	32.71	29.48	0.5210	0.2107	0.1858	35.66	59.56	4.78	2.22	1.56	0.65	29.43
	50.17	60.74	30.47	7.55	32.45	26.21	0.5190	0.1963	0.1733	33.39	62.18	4.43	1.98	1.44	0.54	27.32
GNR 10	50.45	60.74	30.64	7.50	35.18	60.47	0.5018	0.2202	0.1699	33.86	56.12	10.02	4.54	3.53	1.01	22.25
	50.03	60.74	30.39	7.52	35.75	71.31	0.5028	0.2353	0.1744	34.69	53.20	12.11	5.36	4.33	1.03	19.27
GNR 11	50.29	60.74	30.55	22.52	39.87	41.41	0.5115	0.3334	0.2976	58.18	34.82	7.00	9.32	2.79	6.53	70.07
	50.73	60.74	30.81	22.51	39.29	37.66	0.5203	0.3255	0.2869	55.14	37.44	7.42	8.48	2.91	5.56	65.61
GNR 12	51.03	60.74	31.00	22.55	50.74	87.53	0.5036	0.3521	0.2607	51.77	30.08	18.15	19.74	9.21	10.53	53.35
	50.68	60.74	30.78	22.52	49.62	83.64	0.5045	0.3263	0.2436	48.29	35.32	16.39	18.84	8.13	10.70	56.82
GNR 13	50.41	60.74	30.62	7.51	32.71	27.83	0.5074	0.2489	0.2300	45.33	50.95	3.72	2.09	1.22	0.87	41.69
	50.84	60.74	30.88	7.50	32.90	26.96	0.5106	0.2358	0.2161	42.32	53.82	3.86	2.02	1.27	0.75	37.24
GNR 14	51.12	60.74	31.05	7.50	34.81	50.14	0.5169	0.2244	0.1826	35.33	56.59	8.09	3.76	2.81	0.95	25.14
	50.34	60.74	30.58	7.50	35.38	63.98	0.5201	0.2156	0.1671	32.13	58.55	9.33	4.80	3.30	1.50	31.28
GNR 15	50.59	60.74	30.73	22.51	43.11	55.01	0.5121	0.4399	0.3835	74.89	14.10	11.01	12.38	4.75	7.63	61.65
	50.49	60.74	30.67	22.50	41.71	49.06	0.5141	0.4238	0.3634	70.69	17.56	11.75	11.04	4.90	6.14	55.62
GNR 16	50.11	60.74	30.44	22.50	46.01	69.21	0.5231	0.4287	0.3572	68.29	18.05	13.67	15.57	6.29	9.28	59.61
	50.38	60.74	30.60	22.50	45.34	65.52	0.5194	0.4197	0.3453	66.48	19.20	14.32	14.74	6.49	8.25	55.94

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-5 The average of %GNR, %free NR, %free PMMA, %GE and %conversion obtained from 2^4 factorial design

Exp.	Ave. %GNR	Ave. %Free NR	Ave. %Free PMMA	Ave. %GE	Ave. %Conversion
GNR 01	37.2	57.1	5.7	39.3	42.3
GNR 02	33.4	55.5	11.1	16.8	62.6
GNR 03	56.6	29.9	13.5	55.0	59.0
GNR 04	44.2	36.8	19.0	53.8	95.3
GNR 05	39.8	55.1	5.1	33.4	34.2
GNR 06	38.1	55.7	6.2	15.3	32.4
GNR 07	65.5	24.3	10.2	67.7	62.7
GNR 08	68.7	14.2	17.1	48.0	66.5
GNR 09	34.5	60.9	4.6	28.4	27.8
GNR 10	34.3	54.7	11.1	20.8	65.9
GNR 11	56.7	36.1	7.2	67.8	39.5
GNR 12	50.0	32.7	17.3	55.1	85.6
GNR 13	43.8	52.4	3.8	39.5	27.4
GNR 14	33.7	57.6	8.7	28.2	57.1
GNR 15	72.8	15.8	11.4	58.6	52.0
GNR 16	67.4	18.6	14.0	57.8	67.4

Table D-6 Univariate data for graft copolymerization of MMA onto NR induced by microwave irradiation at 60°C

Exp.	NR (g)	%DRC	NR Content (g)	MMA (g)	Graft Product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
INT																
GNR_I01	50.77	60.74	30.84	15.11	39.50	57.33	0.5335	0.4155	0.3812	71.45	22.12	6.43	8.66	2.54	6.12	70.68
	50.65	60.74	30.77	15.03	39.84	60.37	0.5248	0.4201	0.3795	72.31	19.95	7.74	9.07	3.08	5.99	66.03
GNR_I02	50.28	60.74	30.54	15.06	44.50	92.71	0.5324	0.4451	0.4016	75.43	16.40	8.17	13.96	3.64	10.33	73.96
	50.58	60.74	30.72	15.08	44.85	93.68	0.5289	0.4395	0.3915	74.02	16.90	9.08	14.13	4.07	10.06	71.19
GNR_I03	50.54	60.74	30.70	15.01	42.74	80.21	0.5085	0.3971	0.3390	66.67	21.91	11.43	12.04	4.88	7.16	59.44
	50.24	60.74	30.52	15.12	41.89	75.22	0.5105	0.3854	0.3257	63.80	24.51	11.69	11.37	4.90	6.47	56.93
GNR_I04	50.04	60.74	30.40	15.01	43.63	88.16	0.5063	0.3401	0.2705	53.43	32.83	13.75	13.23	6.00	7.24	54.68
	50.32	60.74	30.56	15.13	44.05	89.13	0.5105	0.3428	0.2725	53.38	32.85	13.77	13.49	6.07	7.42	55.02
GNR_I05	50.55	60.74	30.70	15.04	40.90	67.81	0.5248	0.3459	0.2638	50.27	34.09	15.64	10.20	6.40	3.80	37.24
	50.68	60.74	30.78	15.11	41.25	69.26	0.5156	0.3348	0.2586	50.16	35.07	14.78	10.47	6.10	4.37	41.75
MMA																
GNR_M01	50.71	60.74	30.80	7.54	38.27	99.04	0.5501	0.4658	0.4163	75.68	15.32	9.00	7.47	3.44	4.03	53.90
	50.50	60.74	30.67	7.54	37.88	95.61	0.5512	0.4713	0.4175	75.74	14.50	9.76	7.21	3.70	3.51	48.69
GNR_M02	50.51	60.74	30.68	12.01	39.99	77.55	0.5393	0.4680	0.4238	78.58	13.22	8.20	9.31	3.28	6.03	64.80
	50.36	60.74	30.59	12.06	40.15	79.30	0.5140	0.4511	0.4151	80.76	12.24	7.00	9.56	2.81	6.75	70.59

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-6 Univariate data for graft copolymerization of MMA onto NR induced by microwave irradiation at 60°C (continued)

Exp.	NR (g)	%DRC	NR Content (g)	MMA (g)	Graft Product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
GNR_M03	50.54	60.74	30.70	15.01	42.74	80.21	0.5085	0.3971	0.3390	66.67	21.91	11.43	12.04	4.88	7.16	59.44
	50.24	60.74	30.52	15.12	41.89	75.22	0.5105	0.3854	0.3257	63.80	24.51	11.69	11.37	4.90	6.47	56.93
GNR_M04	50.26	60.74	30.53	18.01	47.52	94.35	0.5202	0.4073	0.3131	60.19	21.70	18.11	16.99	8.61	8.39	49.35
	50.37	60.74	30.59	18.32	47.08	89.99	0.5282	0.3948	0.3097	58.63	25.26	16.11	16.49	7.59	8.90	53.99
GNR_M05	50.23	60.74	30.51	22.53	43.38	57.10	0.5386	0.4011	0.3024	56.15	25.53	18.33	12.87	7.95	4.92	38.22
	50.63	60.74	30.75	22.56	44.53	61.08	0.5183	0.4035	0.2956	57.03	22.15	20.82	13.78	9.27	4.51	32.73
MWP																
GNR_P01	50.47	60.74	30.65	15.08	41.2	69.92	0.5285	0.2652	0.1904	36.03	49.82	14.15	10.55	5.83	4.71	44.71
	50.50	60.74	30.68	15.03	42.05	75.70	0.5196	0.2568	0.1878	36.14	50.58	13.28	11.37	5.58	5.79	50.91
GNR_P02	50.11	60.74	30.43	15.02	42.55	80.66	0.5426	0.3578	0.3198	58.94	34.06	7.00	12.12	2.98	9.14	75.41
	50.02	60.74	30.38	15.03	43.08	84.49	0.5218	0.3347	0.3012	57.72	35.86	6.42	12.70	2.77	9.93	78.22
GNR_P03	50.54	60.74	30.70	15.01	42.74	80.21	0.5085	0.3971	0.3390	66.67	21.91	11.43	12.04	4.88	7.16	59.44
	50.24	60.74	30.52	15.12	41.89	75.22	0.5105	0.3854	0.3257	63.80	24.51	11.69	11.37	4.90	6.47	56.93
GNR_P04	50.06	60.74	30.41	15.04	43.38	86.30	0.4983	0.3889	0.3210	64.42	21.95	13.63	12.97	5.91	7.06	54.44
	50.16	60.74	30.47	15.03	44.42	92.85	0.5215	0.4058	0.3245	62.22	22.19	15.59	13.95	6.92	7.03	50.37
GNR_P05	50.32	60.74	30.56	15.06	41.94	75.57	0.5208	0.3807	0.3019	57.97	26.90	15.13	11.38	6.35	5.03	44.23
	50.23	60.74	30.51	15.02	42.07	76.94	0.5216	0.3795	0.2991	57.34	27.24	15.41	11.56	6.48	5.07	43.90

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-6 Univariate data for graft copolymerization of MMA onto NR induced by microwave irradiation at 60°C (continued)

Exp.	NR (g)	%DRC	NR Content (g)	MMA (g)	Graft Product (g)	% Conv.	Sample (g)	Wt.A (g)	Wt.B (g)	%GNR	%Free NR	%Free PMMA	Total MMA (g)	Free PMMA (g)	Grafted MMA (g)	%GE
ET																
GNR_T01	50.42	60.74	30.62	15.10	40.52	65.54	0.5264	0.2831	0.2005	38.09	46.22	15.69	9.90	6.36	3.54	35.75
	50.57	60.74	30.72	15.02	39.56	58.88	0.5215	0.2756	0.1996	38.27	47.15	14.57	8.84	5.77	3.08	34.82
GNR_T02	50.82	60.74	30.87	15.05	41.25	69.01	0.5108	0.3146	0.2560	50.12	38.41	11.47	10.38	4.73	5.65	54.43
	50.13	60.74	30.45	15.15	40.56	66.77	0.5325	0.3356	0.2686	50.44	36.98	12.58	10.11	5.10	5.01	49.54
GNR_T03	50.54	60.74	30.70	15.01	42.74	80.21	0.5085	0.3971	0.3390	66.67	21.91	11.43	12.04	4.88	7.16	59.44
	50.24	60.74	30.52	15.12	41.89	75.22	0.5105	0.3854	0.3257	63.80	24.51	11.69	11.37	4.90	6.47	56.93
GNR_T04	50.08	60.74	30.42	15.02	44.09	91.01	0.4983	0.3889	0.3210	64.42	21.95	13.63	13.67	6.01	7.66	56.05
	50.24	60.74	30.51	15.37	43.86	86.84	0.5148	0.3902	0.3187	61.91	24.20	13.89	13.35	6.09	7.25	54.36
GNR_T05	50.31	60.74	30.56	15.01	40.93	69.08	0.5326	0.3772	0.3154	59.22	29.18	11.60	10.37	4.75	5.62	54.20
	50.46	60.74	30.65	15.29	41.15	68.69	0.5263	0.3744	0.3105	59.00	28.86	12.14	10.50	5.00	5.51	52.42

Wt.A = Graft product was extracted by light petroleum ether for 24 h.

Wt.B = Graft product was extracted by light petroleum ether for 24 h. and then extracted by acetone for 24 h.

Table D-7 Univariate average data for graft copolymerization of MMA on to NR induced by microwave irradiation at 60°C

Exp.	INT	MMA	MWP	ET	Grafting properties			%GE	%Conversion
					%Graft	%Free	%Free		
	(phr)	(phr)	(W)	(min)	NR	NR	PMMA		
GNR I01	**0.5**	50	100	20	71.9	21.0	7.1	68.4	58.9
GNR I02	**1.0**	50	100	20	74.7	16.7	8.6	72.6	93.2
GNR I03	**1.5**	50	100	20	65.2	23.2	11.6	58.2	77.7
GNR I04	**2.0**	50	100	20	53.4	32.8	13.8	54.8	88.6
GNR I05	**2.5**	50	100	20	50.2	34.6	15.2	39.5	68.5
GNR M01	1.5	**25**	100	20	75.7	14.9	9.4	51.3	97.3
GNR M02	1.5	**40**	100	20	79.7	12.7	7.6	67.7	78.4
GNR M03	1.5	**50**	100	20	65.2	23.2	11.6	58.2	77.7
GNR M04	1.5	**60**	100	20	59.4	23.5	17.1	51.7	92.2
GNR M05	1.5	**75**	100	20	56.6	23.8	19.6	35.5	59.1
GNR P01	1.5	50	**50**	20	36.1	50.2	13.7	47.8	72.8
GNR P02	1.5	50	**75**	20	58.3	35.0	6.7	76.8	82.6
GNR P03	1.5	50	**100**	20	65.2	23.2	11.6	58.2	77.7
GNR P04	1.5	50	**125**	20	63.3	22.1	14.6	52.4	89.6
GNR P05	1.5	50	**150**	20	57.7	27.1	15.3	44.1	76.3
GNR T01	1.5	50	100	**5**	38.2	46.7	15.1	35.3	62.2
GNR T02	1.5	50	100	**10**	50.3	37.7	12.0	52.0	67.9
GNR T03	1.5	50	100	**20**	65.2	23.2	11.6	58.2	77.7
GNR T04	1.5	50	100	**30**	63.2	23.1	13.8	55.2	88.9
GNR T05	1.5	50	100	**40**	59.1	29.0	11.9	53.3	68.9

APPENDIX E

Calculation of %conversion, %grafting properties and %GE

From univariate experiment, all obtained data for GNR_P02 #1 are:

Weight of NR latex **(A)**	= 50.11 g
NR content (from %DRC = 60.74) **(B)**	= 30.43 g
Weight of MMA charged **(C)**	= 15.02 g
Weight of graft product **(D)**	= 42.55 g
Weight of sample **(E)**	= 0.5426 g
Weight of sample after extraction with PE **(F)**	= 0.3578 g
Weight of sample after extraction with acetone **(G)**	= 0.3198 g

1. %Conversion

$$\%\text{Conversion} = (D - B)/C \times 100$$
$$= (42.55 - 30.43)/15.02 \times 100$$
$$= 80.69$$

2. %Graft natural rubber, GNR

$$\%\text{GNR} = G/E \times 100$$
$$= 0.3198/0.5426 \times 100$$
$$= 58.94$$

3. %Free NR

$$\%\text{Free NR} = (E - F)/E \times 100$$
$$= (0.5426 - 0.3578)/0.5426 \times 100$$
$$= 34.06$$

4. %Free poly(methyl methacrylate), PMMA

$$\%\text{Free PMMA} = (F - G)/E \times 100$$
$$= (0.3578 - 0.3198)/0.5426 \times 100$$
$$= 7.00$$

5. Total MMA

Total MMA = D – B

= 42.55 – 30.43

= 12.12 g

6. Free PMMA

Free PMMA = (%free PMMA/100) × D

= (7.0/100) × 42.55

= 2.98 g

7. Grafted MMA

Grafted MMA = Total MMA − Free PMMA

= 12.12 – 2.98

= 9.14 g

8. %Grafting efficiency, GE

%GE = (Grafted MMA/Total MMA) × 100

= (9.14/12.12) × 100

= 75.41

APPENDIX F

Calculation of 2^4 factorial design experiments

Table F-1 Contrast constants, effect estimate and sum of squares of %GNR for the 2^4 factorial design

Experiment	Run label	Factorial effect															
		A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	%GNR
1	(1)	-1	-1	1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	1	37.22
2	*a*	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	1	-1	-1	33.41
3	*b*	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1	56.62
4	*ab*	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1	44.18
5	*c*	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1	39.76
6	*ac*	1	-1	-1	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1	38.06
7	*bc*	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	65.51
8	*abc*	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	68.68
9	*d*	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	1	-1	34.53
10	*ad*	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	1	1	34.27
11	*bd*	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	56.66
12	*abd*	1	1	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	50.03
13	*cd*	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	43.83
14	*acd*	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	33.73
15	*bcd*	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	72.79
16	*abcd*	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	67.38
Contrast		-37.17	187.04	-5.45	82.81	9.11	50.93	24.57	9.76	-7.62	13.97	2.08	1.67	-26.34	-1.47	-2.42	$\bar{x}$ = 48.54
Effect Estimate		-4.65	23.38	-0.68	10.35	1.14	6.37	3.07	1.22	-0.95	1.75	0.26	0.21	-3.29	-0.18	-0.30	
Sum of squares		86.34	2186.49	1.86	428.58	5.19	162.11	37.73	5.95	3.63	12.20	0.27	0.17	43.36	0.14	0.37	
																Σ Sum of squares =	2974.37

Table F-2 Contrast constants, effect estimate and sum of squares of %free NR for the 2^4 factorial design

Experiment	Run label	Factorial effect															
		A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	%free NR
1	(1)	-1	-1	1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	1	57.1
2	*a*	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	1	-1	-1	55.5
3	*b*	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1	29.9
4	*ab*	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1	36.8
5	*c*	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1	55.1
6	*ac*	1	-1	-1	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1	55.7
7	*bc*	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	24.3
8	*abc*	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	14.2
9	*d*	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	1	-1	60.9
10	*ad*	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	1	1	54.7
11	*bd*	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	36.1
12	*abd*	1	1	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	32.7
13	*cd*	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	52.4
14	*acd*	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	57.6
15	*bcd*	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	15.8
16	*abcd*	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	18.6
Contrast		-5.70	-240.48	-1.84	-69.88	2.80	-55.20	-24.32	0.16	2.37	-3.90	2.61	-10.04	32.41	-2.40	13.96	41.09
Effect Estimate		-0.71	-30.06	-0.23	-8.74	0.35	-6.90	-3.04	0.02	0.30	-0.49	0.33	-1.25	4.05	-0.30	1.75	
Sum of squares		2.03	3614.54	0.21	305.21	0.49	190.43	36.96	0.00	0.35	0.95	0.43	6.30	65.66	0.36	12.19	
																Σ Sum of squares =	4236.10

Table F-3 Contrast constants, effect estimate and sum of squares of %free PMMA for the 2^4 factorial design

Experiment	Run label	Factorial effect															
		A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	%free PMMA
1	(1)	-1	-1	1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	1	5.7
2	*a*	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	1	-1	-1	11.1
3	*b*	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1	13.5
4	*ab*	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1	19.0
5	*c*	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1	5.1
6	*ac*	1	-1	-1	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1	6.2
7	*bc*	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	10.2
8	*abc*	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	17.1
9	*d*	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	1	-1	4.6
10	*ad*	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	1	1	11.1
11	*bd*	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	7.2
12	*abd*	1	1	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	17.3
13	*cd*	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	3.8
14	*acd*	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	8.7
15	*bcd*	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	11.4
16	*abcd*	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	14.0
Contrast		42.86	53.44	7.29	-12.93	-11.92	4.27	-0.25	-9.92	5.24	-10.07	-4.69	8.37	-6.07	3.88	-11.54	10.37
Effect Estimate		5.36	6.68	0.91	-1.62	-1.49	0.53	-0.03	-1.24	0.66	-1.26	-0.59	1.05	-0.76	0.48	-1.44	
Sum of squares		114.83	178.52	3.32	10.45	8.88	1.14	0.00	6.15	1.72	6.34	1.37	4.38	2.31	0.94	8.33	
																Σ Sum of squares =	348.67

Table F-4 Contrast constants, effect estimate and sum of squares of %GE for the 2^4 factorial design

Experiment	Run label	Factorial effect															
		A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	%GE
1	(1)	-1	-1	1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	1	39.3
2	*a*	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	1	-1	-1	16.8
3	*b*	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1	55.0
4	*ab*	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1	53.8
5	*c*	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1	33.4
6	*ac*	1	-1	-1	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1	15.3
7	*bc*	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	67.7
8	*abc*	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	48.0
9	*d*	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	1	-1	28.4
10	*ad*	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	1	1	20.8
11	*bd*	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	67.8
12	*abd*	1	1	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	55.1
13	*cd*	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	39.5
14	*acd*	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	28.2
15	*bcd*	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	58.6
16	*abcd*	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	57.8
Contrast		-93.93	242.14	25.04	11.57	-5.88	-10.73	-7.25	26.98	28.96	2.93	-14.53	12.49	22.40	-39.40	38.33	42.83
Effect Estimate		-11.74	30.27	3.13	1.45	-0.74	-1.34	-0.91	3.37	3.62	0.37	-1.82	1.56	2.80	-4.92	4.79	
Sum of squares		551.38	3664.42	39.20	8.36	2.16	7.19	3.29	45.49	52.43	0.54	13.19	9.75	31.35	97.00	91.84	
																Σ Sum of squares =	4617.59

VITA

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