

## THE MANUFACTURE OF GLOVES USING RVNRL: PARAMETERS OF THE COAGULANT DIPPING PROCESS

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(Received: May 13, 1998; Accepted: August 26, 1998)

**Abstract** - Surgical gloves were manufactured using the RVNRL process. A fractional factorial design at two levels showed that five parameters of the coagulant dipping process which were studied independent. Coagulant concentration and dwell time in the radiovulcanized latex presented major main effects while the temperature of the former before dipping into the radiovulcanized latex and the flow time of the radiovulcanized latex on the former surface presented opposite main effects. The withdrawal rate of the former from the radiovulcanized latex did not change glove thickness. The mathematical correlation between the estimates of thickness and the significant main effects of coded variables was  $\hat{y} = 0.212 + 0.025x_1 + 0.019x_2$ . This optimized equation allowed reproduction of a surgical glove thickness in the range of 0.157 to 0.291mm, which is considered acceptable by international standard specification.

**Keywords:** Surgical gloves manufacture, radiation vulcanization, natural rubber latex, coagulant dipping process, factorial design.

## INTRODUCTION

Rubber goods can be manufactured from vulcanized natural rubber latex by five different processes: dipping, casting, clothing, extruding and foaming. The use of latex concentrate for making dipped articles may be the oldest and simplest process in latex technology.

There are three latex dipping processes: straight dipping, coagulant dipping and heat-sensitive dipping. The coagulant dipping process is the most commonly used process. The former is dipped into the coagulant solution, withdrawn and dipped into the latex compound during a suitable dwell time. The former is then slowly withdrawn, inverted and dried. Thickness is about  $0.15-0.30 \times 10^{-3}\text{m}$ .

Articles manufactured by the vulcanized latex process used throughout the world release air pollutants such as  $\text{SO}_x$ ,  $\text{NO}_x$  and  $\text{ZnO}$  by incineration and cause dermatitis by contact because sulfur is cytotoxic (Makuuchi, 1990; Nakamura et al., 1990). In addition, small quantities of dithiocarbamates are transformed into carcinogenic nitrosamines causing environmental and rubber goods contamination during conventional vulcanization (Makuuchi, 1992).

An alternative vulcanization process, the radiation vulcanization of natural rubber latex (RVNRL), was developed in the 1980s (Machi, 1990; Utama, 1990). The vulcanization dose is about 200kGy but the sensitizer reduces for only 8-10kGy and RVNRL became an industrial process (Aroonvisoot and Makuuchi, 1990; Zhonghai and Makuuchi, 1990). The RVNRL process is completely free of nitrosamines. The absence of dithiocarbamates, which also function as antioxidants and cytotoxins, facilitates easy degradation of rubber goods in the environment. Toy and advertising balloons manufactured by RVNRL process degrade quickly in the ground. These rubber goods have a low cytotoxicity and do not produce environmental damage (Makuuchi, 1992). They are more transparent (98%) and softer (Shimamura, 1990). Their mechanical properties are similar to those of articles manufactured by the conventional process (Makuuchi, 1990). Currently surgical gloves and others medical supplies are being manufactured by RVNRL process in Japan and Asia (Ridwan, 1985; Siri-Upathum and Chvacharenpun, 1992; Zin, 1997).

Gorton, and Iyer (Gorton, 1967; Gorton and Iyer, 1973) developed the rubber deposition theory and showed the

dependency of thickness on viscosity, dwell time and coagulant solution for the conventional vulcanization process which has two steps, one of sulfur prevulcanization of latex using solid agent dispersion and the other of sulfur postvulcanization of the article. In the RVNRL process vulcanization occurs in only one irradiation step in the presence of liquid sensitizer dispersion at room temperature.

**Table 1: Latex formulation**

<b>Component</b>	<b>Weight (kg)</b>
Latex	123.78
H <sub>2</sub> O (50% of total solid contents)	2.47
2/3 of 10% KOH (0.2 phr)	1.00
Sensitizer emulsion	22.75
n-butyl acrylate (3phr)	2.19
H <sub>2</sub> O	19.96
1/3 of 10% KOH (0.2 phr)	0.50
emulsifier (1%)	0.23
<b>Formulated latex</b>	<b>150.00</b>
Antioxidant emulsion (50%)	1.46
Irganox 1520 (1phr)	0.73
H <sub>2</sub> O	0.67
emulsifier (1%)	0.02
<b>Radiovulcanized latex</b>	<b>151.46</b>

Gazeley and Pendle (1990) studied the interdependency of film thickness and dwell time in the vulcanized latex containing n-butyl acrylate (n-BA) as the sensitizer. The coagulant was Ca(NO<sub>3</sub>)<sub>2</sub>. In Thailand Siri-Upathum and

Chvacharernpun (1992) developed a pilot plant for RVNRL surgical gloves which had a capacity of 2000-3000kg per batch and used 2-ethylhexylacrylate/ $\text{CCl}_4$  as the sensitizer. Two parameters of the coagulant dipping process were established as optimum: total solids in the range of 50-52% and a dwell time of 50s associated with a partial dry at 363K. However, in none of these studies was the factorial design used to evaluate the interdependency of variables or improve this industrial process. This statistical technique can be applied to experimental designs in which data will be collected (Box et al., 1978; Barros Neto et al., 1995). One of its advantages is the possibility of obtaining more information about many processing variables when they are studied simultaneously in relatively few experiments. Another advantage is that it allows the improvement of the process (Burton and Nickless, 1987).

This paper reports the effects of five processing parameters of coagulant dipping on the thickness of surgical gloves manufactured by RVNRL using factorial design. This practical pilot plant application of RVNRL process addresses the industrial manufacture of rubber goods.

## **EXPERIMENTAL**

### **Chemicals**

Commercial concentrated latex, with a total solids content of 60.97% and a dry rubber content of 58.97%, has a pH of 10.08 and a density of  $0.98 \text{ kg/dm}^3$  at room temperature (ASTM 1076-79). Water was purified by passing through ion exchange columns. Industrial grade HCl and NaOH were used for cleaning the porcelain former. Analytical grade 10% KOH was used as the latex stabilizer. Analytical grade n-BA was used as the sensitizer in a 50% emulsion. 1phr of 50% commercial antioxidant, Irganox 1520, was added to the irradiated latex as an emulsion. The commercial emulsifier, Emulvin, was used as covalent kind. Analytical grade HCl and  $\text{CaCO}_3$  in powder form were used to prepare the coagulant solution.

### **Formulation**

Formulation of latex (Machi, 1990; Souza, 1994; Canavel, 1993) using 3phr n-BA / 0.2phr KOH as the sensitizer ([Table 1](#)) was as follows: the total solids content was reduced to

50%, 2/3 of the 10% KOH solution were added to the latex and after 300s the n-BA emulsion, which has a 1% emulsifier and 1/3 of the 10% KOH solution, was added. The latex was gently stirred at 0.66rps and at room temperature during formulation in a drum measuring 0.2 m<sup>3</sup>. The turbine stirrer was supplied a helix with a diameter of 0.3m. 150kg of latex were kept at room temperature for one hour and stored in three 0.05m<sup>3</sup> plastic containers during four hours. The viscosity of 147.7x10<sup>3</sup> kg/s m was determined at 298K using the Brookfield Viscometer LVF model and spindle 1.

### **Irradiation**

The formulated latex was irradiated without stirring at room temperature using gamma rays from a <sup>60</sup>Co source, an industrial multipurpose source with carrier system design. The dose rate was 1.25 kGy/s. The vulcanization dose was 10kGy. The radiovulcanized latex was kept for 10 days after which time 1phr of 50% antioxidant was added and it was kept for another day.

CaCl<sub>2</sub> was used as the coagulant in the manufacture of the surgical gloves. The 20% CaCl<sub>2</sub>, with pH = 6.26, was obtained from an industrial reaction between 0.035kg of 37% HCl and a stoichiometric excess of CaCO<sub>3</sub> in powder form (equation 1).



### **Coagulant Dipping Parameters**

The experiments were planned according to a fractional factorial design at two levels,  $2^{s-1}_v$  (Box, 1978). In this design the response variables studied are represented by capital letters (A,B,...) and the coded variables by an alpha-numerical set (x<sub>1</sub>, x<sub>2</sub>,...).

The five variables and their maximum and minimum levels ([Table 2](#)) were selected according to information from the international literature (Sundardi and Marga, 1990) and national factories (Contim, 1994; Youssef, 1994). The experimental variables selected and their symbols and units are:

**A** - Coagulant concentration, %

**B** - Former temperature dipped into the radiovulcanized latex, K

**C** - Coagulant flow time on the former surface, s

**D** - Dwell time of former dipped into the radiovulcanized latex, s

**E** - Withdrawal rate of former from the radiovulcanized latex, m/s

[Table 3](#) shows the fractional factorial design  $2^{5-1}_v$  which has 16 different experimental conditions for surgical glove manufacture in Yates order for the five real variables (A, B, C, D, E) and their coded variables ( $x_1, x_2, x_3, x_4, x_5$ ), respectively. This factorial design was carried out randomly order and without replicates. The thickness of each pair of gloves manufactured under 16 experimental conditions was measured in the upper fist, central and palm regions. The thickness for each experimental condition was the mean of the six measurements carrying out with a vertical micrometer ( $0.001 \times 2 \times 10^{-3}$  m).

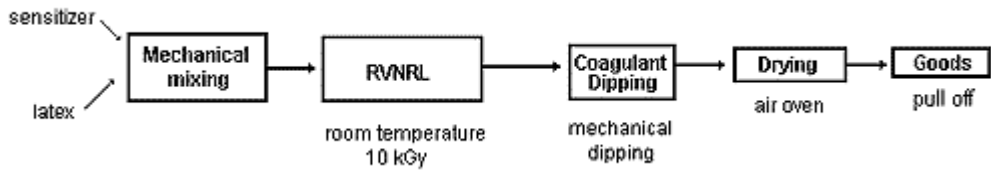
### **Surgical Gloves Manufacture**

Surgical gloves were manufactured by the coagulant dipping process ([Figure 1](#)) (Utama, 1990; Sundardi and Marga, 1990) using a small factory facility. The equipment utilized was a mechanical dipping system ([Figure 2](#)), an industrial electric oven with internal air circulation and a chronometer for controlling the processing variables.

**Table 2: Range of experimental variables**

Variable	Coded levels	
	minimum (-1)	maximum (+1)
<b>A</b> - CaCl <sub>2</sub> concentration, %	8.0	19.7
<b>B</b> - Temperature of former dipped into the latex, K	303	353
<b>C</b> - Coagulant flow time, s.	3	10
<b>D</b> - Dwell time of former in the latex, s	3	12

<b>E</b> - Rate of withdrawal from latex, m/s	0.025	0.10
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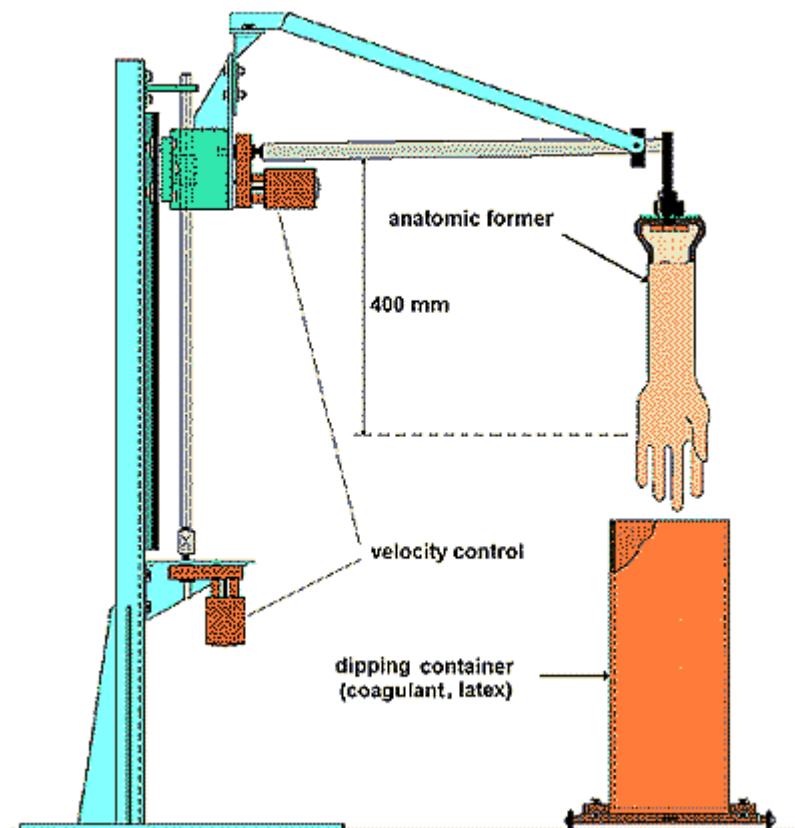
**Figure 1:** Scheme of rubber goods manufactured by coagulant dipping.

**Table 3:** Fractional factorial design matrix,  $2^{5-1}$

Yates Order	Coded variable <sup>a</sup>				
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	X <sub>5</sub>
1	-1	-1	-1	-1	+1
2	+1	-1	-1	-1	-1
3	-1	+1	-1	-1	-1
4	+1	+1	-1	-1	+1
5	-1	-1	+1	-1	-1
6	+1	-1	+1	-1	+1
7	-1	+1	+1	-1	+1
8	+1	+1	+1	-1	-1
9	-1	-1	-1	+1	-1
10	+1	-1	-1	+1	+1
11	-1	+1	-1	+1	+1
12	+1	+1	-1	+1	-1
13	-1	-1	+1	+1	+1
14	+1	-1	+1	+1	-1

15	-1	+1	+1	+1	-1
16	+1	+1	+1	+1	+1

<sup>a</sup>  $x_1, x_2, x_3, x_4, x_5$  are coded variables of A, B, C, D, E respectively.



**Figure 2:** Mechanical dipping system.

Both porcelain formers were treated with 10% caustic soda and 8% HCl and dipped into circulating water to remove the acid and dried for 20 minutes at 253K before the manufacture of each new glove. Then the former was dipped into the coagulant solution for the controlled dwell time at the controlled dipping rate. The excess solution was eliminated when the former was maintained in a vertical position during a controlled time and then dried in an air oven for 15 minutes. Immediately thereafter it was dipped into the radiation vulcanized latex for the controlled dwell



time and dipping rate. The glove fist edging was made by rolling for  $1-2 \times 10^{-2}$  m. The molded radiovulcanized latex was dried in the air oven to control for time and temperature. The glove was pulled off the former using talc.

## RESULTS

In the RVNRL process of surgical gloves manufacture, the coagulant dipping step was studied to evaluate which factors had the significant effects on glove thickness in order to develop a more economical process.

The mean thickness ( $y$ ) for each of the 16 experimental conditions of the fractional factorial design  $2_{V}^{5-1}$  (Table 4) permitted a study of the principal effects of each real variable and their interdependencies. The STATGRAPH statistical software was used for calculating the effects (Table 5).

In the significance analysis of effects by the normal probability plotting, the negligible effects are normally distributed around zero (Box et. al., 1978). They would therefore be plotted on a normal probability graph paper as a straight line passing in (0,0) point. The significant effects have means different from zero and they are positioned off this straight line. The effects of  $x_1$  (0.050),  $x_2$  (0.016),  $x_3$  (-0.016) and  $x_4$  (0.038) were considerable, while that of  $x_5$  (0.001) was negligible (Figure 3).

**Table 4: Results of fractional factorial design,  $2_{V}^{5-1}$**

Order <sup>1</sup>		Real variable <sup>2</sup>					Coded variable <sup>3</sup>					$y^4$ (mm)
Y	R	A	B	C	D	E	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	X <sub>5</sub>	
1	14	8	303	3	3	0.10	-1	-1	-1	-1	+1	0.165
2	5	19.7	303	3	3	0.025	+1	-1	-1	-1	-1	0.214
3	9	8	353	3	3	0.025	-1	+1	-1	-1	-1	0.183
4	2	19.7	353	3	3	0.10	+1	+1	-1	-1	+1	0.233
5	15	8	303	10	3	0.025	-1	-1	+1	-1	-1	0.157

6	8	19.7	303	10	3	0.10	+1	-1	+1	-1	+1	0.207
7	10	8	353	10	3	0.10	-1	+1	+1	-1	+1	0.175
8	1	19.7	353	10	3	0.025	+1	+1	+1	-1	-1	0.215
9	16	8	303	3	12	0.025	-1	-1	-1	+1	-1	0.205
10	6	19.7	303	3	12	0.10	+1	-1	-1	+1	+1	0.250
11	12	8	353	3	12	0.10	-1	+1	-1	+1	+1	0.223
12	3	19.7	353	3	12	0.025	+1	+1	-1	+1	-1	0.291
13	13	8	303	10	12	0.10	-1	-1	+1	+1	+1	0.199
14	7	19.7	303	10	12	0.025	+1	-1	+1	+1	-1	0.238
15	11	8	353	10	12	0.025	-1	+1	+1	+1	-1	0.192
16	4	19.7	353	10	12	0.10	+1	+1	+1	+1	+1	0.254

<sup>1</sup>Y = Yates order, R = order of runs.

<sup>2</sup>A = CaCl<sub>2</sub> concentration (%); B = temperature of former dipped into the radiovulcanized latex (K); C = coagulant flow time (s); D = dwell time of the former dipped into the radiovulcanized latex (s); E = rate of withdrawal from radiovulcanized latex (m/s).

<sup>3</sup>x<sub>1</sub>, x<sub>2</sub>, x<sub>3</sub>, x<sub>4</sub>, x<sub>5</sub> are coded variables of A, B, C, D, E, respectively

<sup>4</sup>Mean thickness value.

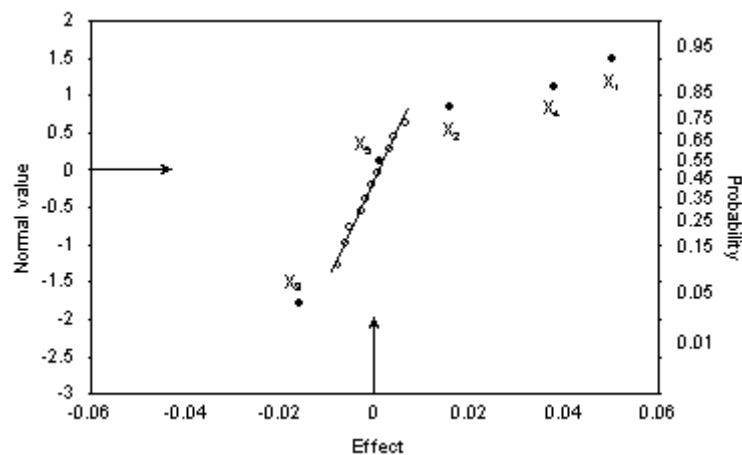
**Table 5: Estimates of effects**

Effect <sup>1</sup>	Estimate
y <sup>2</sup>	0.212
A	0.050
B	0.016
C	-0.016
D	0.038
E	0.001
AB	0.004
AC	-0.002
BC	-0.008
AD	0.003

BD	0.001
CD	-0.006
AE	-0.005
BE	-0.001
CE	0.007
DE	-0.002

<sup>1</sup> A = CaCl<sub>2</sub> concentration (%); B = temperature of former dipped into the radiovulcanized latex (K); C = coagulant flow time (s); D = dwell time of the former dipped into the radiovulcanized latex (s); E = rate of withdrawal from radiovulcanized latex (m/s).

<sup>2</sup> Mean thickness, mm.



**Figure 3:** Significant effect analysis ( $s = 0.035$ ): o - mutual interaction; · - main effect.

The coded variable  $x_1$  had the greatest effect (Figure 3) because thickness increased about 0.05 mm (Table 5) when coagulant concentration was increased from 8% to 19.7% (Table 2). The coded variable  $x_4$  also showed a considerable effect on glove thickness (Figure 3), which increased about 0.038 mm (Table 5) when dwell time was increased 9s (Table 2). The  $x_2$  coded variable, representing temperature of former dipped into the radiovulcanized latex, and  $x_3$  representing flow time of coagulant on former surface after its removal from the coagulant solution, presented effects with equal absolute magnitude but opposites signs (Table

5). The  $x_3$  effect showed a reduction in thickness of 0.016mm, which occurred when the flow time changed from 3 to 10s (Table 2). When the withdrawal time from the coagulant solution was quick, the former drew off the coagulant excess allowing more rubber to be deposited. When the withdrawal time was slower, the coagulant flowed on the former surface. The  $x_4$  effect showed a thickness enhancement of 0.016mm (Table 5) when the former temperature before dipping into the radiovulcanized latex changed from 303 to 353K (Table 2). The thickness was not affected by the withdrawal rate of former from latex when the  $x_5$  coded variable was studied in the range of 0.025 to 0.10 m/s (Table 2).

The ten two-variable interactions did not present any significant effect (Figure 3). The five variables studied were not dependent and enabled a mathematical correlation between the coded variables and thickness to be made by a linear regression model (Table 4). The mathematical equation of the first degree polynomial, adjusted to the experimental values by least squares estimation using the STATGRAPH program, yields the following fitted equation:

$$\hat{Y} = 0.212 + 0.025 x_1 + 0.008 x_2 - 0.008 x_3 + 0.019 x_4 + 0.001 x_5 \quad (2)$$

where  $\hat{Y}$  was the mean value of thickness estimated by this method and  $x_1, x_2, x_3, x_4,$  and  $x_5$  were the coded variables of A, B, C, D, and E, respectively. The regression coefficients are the half of effect values (Table 5) since the latter measures the change of two units from the low level ( $x_i = -1$ ) to the high level ( $x_i = +1$ ) in response to the input variable changes, whereas slope  $b_i$  measures the change in response as  $x_i$  is changed by one unit from  $x_i = 0$  to  $x_i = 1$ .

The significance of variables A (0.000), B (0.005), C (0.005), D (0.000) and E (0.753) (Table 6) also showed that only the E variable was not significant.

In equation 2 the third and fourth terms can be eliminated because the coded variable effects,  $x_2$  and  $x_3$ , were small and opposites when they changed in the range studied proportionally. The last term represented a negligible effect of 0.001. The first y term, 0.212mm, represented the estimate of mean thickness in the experimental interval studied for the model which had a correlation coefficient of

95.7% and an standard error of  $\pm 0.004$ . Therefore equation 2 could be reduced to:

$$\hat{y} = 0.212 + 0.025 x_1 + 0.019 x_4 \quad (3)$$

where  $\hat{y}$  represent the estimate of thickness of surgical gloves in mm and  $x_1$  and  $x_4$  are the coded variables of A and D, respectively. Equation 3 represents the optimization of the RVNRL process and allows the estimation of the thickness of surgical gloves manufactured by the coagulant dipping process. The economic processing parameters ([Table 7](#)) take into account the minor thickness of 0.157mm acceptable by international standard specification (ASTM D 3577-78a).

**Table 6: Regression coefficients of linear model**

Coded variable <sup>1</sup>	Coefficient	s <sup>2</sup>	P <sup>3</sup>
y <sup>4</sup>	0.212	0.002	0.000
x <sub>1</sub>	0.025	0.002	0.000
x <sub>2</sub>	0.008	0.002	0.005
x <sub>3</sub>	-0.008	0.002	0.005
x <sub>4</sub>	0.019	0.002	0.000
x <sub>5</sub>	0.001	0.002	0.753

<sup>1</sup>x<sub>1</sub>, x<sub>2</sub>, x<sub>3</sub>, x<sub>4</sub>, x<sub>5</sub> are coded variables of A, B, C, D, E, respectively.  
<sup>2</sup> Standard deviation. <sup>3</sup> significance level. <sup>4</sup> mean thickness.

**Table 7: Optimized processing parameters for RVNRL surgical gloves manufacture by the coagulant dipping process**

Parameter	Optimized value
CaCl <sub>2</sub> concentration	8%
Temperature of former dipped into the radiovulcanized latex	303K

Coagulant flow time on the former surface	10s
Dwell time of former dipped into the radiovulcanized latex	30s
Rate of withdrawal of former from the radiovulcanized latex	0.10 m/s

## CONCLUSIONS

The fractional factorial design at two levels showed none of the five variable effects studied for the coagulant dipping process depend on the levels of the other four variables. The coagulant concentration and dwell time of the former in the radiovulcanized latex produced a considerable variation in the thickness of the surgical gloves. The mathematical correlation between the coded variables of processing parameters and the thickness estimate ( $\hat{Y}$ ) of surgical gloves manufactured by RVNRL process is

$\hat{Y} = 0.212 + 0.025 x_1 + 0.019 x_4$ . This optimized equation allowed the reproduction of surgical glove thickness in the range of 0.157 to 0.291mm.

## ACKNOWLEDGEMENT

The authors would like to acknowledgment to Issa A. Youssef (in memory) for the fruitful discussions and for allowing us to use the industrial facility.

## NOMENCLATURE

**A** Coagulant concentration, %.

**B** Temperature of former dipped into the radiovulcanized latex, K.

**C** Coagulant flow time on the former surface, s.

**<sup>60</sup>Co** Cobalt 60 radioisotope, half-life = 5.3 years,  $E_g = 2.13 \times 10^{-13}$  and  $1.88 \times 10^{-3}$  J.

**D** Dwell time of former dipped into the radiovulcanized latex, s.

**E** Withdrawal time of former from radiovulcanized latex, s.

**Gy** Gray, absorption dose unit, Gy = 100 rads.

**n-BA** n-Butyl acrylate.

**phr** Per hundred rubber.

**rps** Rotations per second.

**RVNRL** Radiation vulcanized natural rubber latex.

**x<sub>1</sub>** Coded variable of A.

**x<sub>2</sub>** Coded variable of B.

**x<sub>3</sub>** Coded variable of C.

**x<sub>4</sub>** Coded variable of D.

**x<sub>5</sub>** Coded variable of E.

**y** Mean thickness, mm.

$\hat{y}$  Estimate of mean thickness, mm.

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