Progress in Engineering Application and Technology Vol. 4 No. 1 (2023) 39-50
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PEAT

Homepage: http://publisher.uthm.edu.my/periodicals/index.php/peat
e-ISSN : 2773-5303

Optimization of Mould Method for Dipping Process to Produce Antimicrobial and Biodegradable Glove Containing Cassava-Peel & Soy Lecithin as Fillers

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DOI: https://doi.org/10.30880/peat.2023.04.01.004
Received 15 January 2023; Accepted 12 February 2023; Available online 12 June 2023

Abstract: The COVID-19 virus has spread awareness and the demand of biodegradable composite glove is increasing. The objective of this study is to optimize the ratio of fillers and mixing speed in which bio-based fillers loaded glove with good biodegradable and antimicrobial properties can be prepared using dipping method process with Taguchi method performed by using XLSTAT and Microsoft Excel. The cassava-peel starch was acquired by using extraction to be used as bio-based filler along with soy lecithin. The ratio between two fillers and mixing speed were optimized using the design of experiment with Taguchi in which SN Ratio with concept "the bigger the better" have been used. From the data, it was observed after 30 days of soil burial test using SEM, the biodegradable properties in which the surface crack of films prepared were increasing when the cassava-peel filler increased. The mechanical testing shows a great performance of film's tensile strength at 30 phr cassava peel filler, 30 phr soy lecithin filler and 450 rpm mixing speed (NRL3) which conclude that medium amount of fillers were the best for natural rubber latex. By using Taguchi method, the SN ratios shows the A2B2C3 (Cassava-peel filler at 30 phr, Soy lecithin filler at 30 phr and mixing speed at 450 rpm) was the best conditions to prepare biodegradable glove using dipping process. Other conditions also can be further studied in the future such as the vulcanization and pre-vulcanization temperature, maturity period for NRL and mixing time.

Keywords: Cassava-peel starch filler, Soy lecithin filler, Bio-based filler, Biodegradable glove, Taguchi (DoE)

1. Introduction

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Rubber is originated from a milky white liquid known as latex, it is a limited resourse and must be preserved [1]. It is isolated from the plant *Hevea brasiliensis* and other tropical plants such as *Castilloa elastica* [1]. Generally, Natural rubber is hard to be degraded as the bio-degradation process usually takes several years to reach completion [1]. Mass production of rubber-based product especially rubber gloves in pandemic era cause increasing solid waste disposal for landfill [2]. Besides landfills, another way of rubber gloves disposal is by open burning or incineration which is not environmentally friendly [2]. Therefore, the glove disposal issue has attracted the public and authority's attention to seeking a greener and enhanced biodegradability with good anti-microbial properties method to replace current initiation because the drawbacks arise from incineration and open burning [4]. As compared to open burning, biodegradation is more favourable as it has no negative effect on the natural environment at the same time able to aid with nutrient recycling in soil [4].

Cassava and soy are the local ingredient that is available abundantly in Malaysia [3]. It acts as the raw material in the production of snacks and beverages such as crisps and crackers [3]. The rapid development of the food processing industries in Malaysia has generated a large amount of cassava-peel and soy waste as industrial waste every year and they will end up in open burning or illegal dumping [3]. Therefore, there is a potential chance to utilize these wastes instead of throwing them away as they are abundant in large quantities, cheap, biodegradable and renewable resources.

This study is to produce the NRL composite films loaded with cassava-peel and soy lecithin fillers using the blending method and characterize the biodegradability and antimicrobial properties of prepared gloves and analyse the optimisation of dipping methods process used to prepare NRL composite glove containing cassava-peel and soy lecithin by using Taguchi Method.

2. Methodology

This section describes all the necessary information that is required to obtain the result of study.

2.1 Materials and Apparatus

The cassava peel (Manihot talisman) was acquired from the wholesale market at Pandan. High Ammonia Natural rubber latex Concentrate was purchase from Getahindus Sdn. Bhd. Potassium hydroxide (KOH, Mn of 56.11 g/mol), Sodium metabisulfite (Na2S2O5, Mn= 197.11g/mol) were purchased from Merck. 10% of Hydrochloric acid (HCl, conc. = 37%) was purchased from R&M Chemicals. Sodium Hydroxide (NaOH, Mn = 40.00g/mol) was purchased from Sigma-Aldrich. Anchoid, A308 Composite Dispersion were acquired from Aquaspersion Sdn Bhd. Calcium nitrate (Ca (NO3)2, Mn = 164.088 g.mol-1) were acquired from Excelkos Sdn Bhd.

The Laboratory Blender (Waring Commercial, United State.), Analytical balance (A&D Company, Limited, Japan), magnetic stirrer (Corning, United States) and Fume Hood (Esco, Singapore.) are located at the Material Lab under JTKK, FTK UTHM Pagoh. The Oven (Memmert, Germany) and water bath (Daihan Scientific, South Korea) are located at the Upstream Lab under JTKK, FTK, UTHM Pagoh. Fourier Transform Infrared Spectroscopy (Agilent, United States.) is located at the Downstream Bioprocessing Lab under JTKK, FTK, UTHM Pagoh. Instron Universal testing machine (Ametek, United States.) is located at the Textiles Testing Lab under JTKM, FTK UTHM Pagoh.

2.2 Design of Experiment (Taguchi Method) using XLSTAT

The Taguchi method was chosen as an optimisation method to optimise the characteristic of the Natural Rubber Latex with different type of filler, percentage of loading filler, and mixture speed. These three conditions were the factor chosen to be optimised as listed in Table 2.1. The optimisation using the Taguchi method was based on approach "the larger the better". This approach was based on signal and noise ratio (SN) that was calculated in Microsoft Excel with XLSTAT integrated.

Table 2.1: Three levels and three factors in Design of Experiment

Factor/Level	0	1	2
A: Cassava peel filler loading, phr	20	30	40
B: Soy lecithin filler loading, phr	20	30	40
C: Mixing Speed, RPM	350	400	450

2.3 Preparation of samples

The samples were prepared based on conditions generated by design of experiment as shown in Table 2.2 and the NRL mixture was prepared according to the formulation as given in Table 2.3.

Table 2.2: Design of experiment by Taguchi Method

Samples	Cassava peel loading filler(phr)	Soy lecithin filler loading (phr)	Mixing speed (rpm)
NRL 1	40	30	300
NRL 2	20	20	450
NRL 3	30	30	450
NRL 4	20	40	350
NRL 5	40	20	400
NRL 6	30	20	350
NRL 7	30	40	400
NRL 8	40	40	450
NRL 9	20	30	400

Table 2.3: Compounding ingredients for NRL films with different amounts of loading of filler (in phr).

Ingredients	Control NRL	NRL 1	NRL 2	NRL 3	NRL 4	NRL 5	NRL 6	NRL 7	NRL 8	NRL 9
60% NRL	100	100	100	100	100	100	100	100	100	100
A308 Composite Dispersion	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Cassava-peel starch dispersion, phr	-	40	20	30	20	40	30	30	40	20
Soy Lecithin Dispersion, phr	-	30	20	30	40	20	20	40	40	30

All the ingredients were homogenized for two hours using a magnetic stirrer mixing speed as shown at Table 2 and pre-vulcanized at the temperature of 70 °C. The chloroform number test is carried out until chloroform number 2 is obtained [15]. The NRL compound is maturated overnight under room temperature before film preparation. Clean glass plates were dipped in coagulant (10 % calcium nitrate) for 5 seconds and dried in the air oven for 10 minutes at 100°C before being cooled under room temperature for 5 minutes [5]. The glass plates were then used to cast the NRL by using the film applicator. The films cast were cured in the air oven for 20 minutes at 100°C before being cooled under room temperature for 24 hours. The films were then stripped by the aids of cassava powder to prevent the films from sticking together and damaging the surfaces of the films [5].

2.3 Characterization of the NRL films for the morphological analysis, Biodegradation properties and mechanical strength

Morphology Analysis using SEM: The NRL composite films prepared were observed under the Energy-dispersive X-ray spectroscopy Scanning Electron Microscope (SEM) with the model of Hitachi VP-SEM SU1510 to observe the morphological structure present on the surface of the dispersion. All dispersions tested were previously cut into a square size with a dimension of 1cm ×1cm. Then, the prepared samples were inserted into the microscope and the images were observed and captured at 1000x, 5000x and 10000x magnifications [5].

Mechanical properties test: The tensile strength of the NRL films was tested by using the Instron Universal testing machine. The sample was prepared, and the test specification was according to ASTM standard D412-92 method [18]. Dumbbell test pieces of Type 1 were prepared from the NR latex films. The dimension of the Dumbbell test pieces is as followed: The overall length: 115mm, The width of end: 25±1mm, The Length of the narrow portion: 33±2mm. A crosshead speed of 500 mm/min was used. The test was carried out at room temperature. From the tensile data, the tensile stress, young's modulus, and elongation at break were determined [18].

Soil burial test to determine biodegradation properties: The soil burial test was carried out to replicate the real-life natural biodegradation condition of polymers (ASTM International, 2019). The films were buried in controlled soil for 1 month at room temperature [17]. The films were tested after burial for a month by taken out from the soil and clean with distilled water and dried at room temperature overnight [17].

The alteration in the morphologies of film surface after soil burial test was observed using SEM at 1000x, 5000x and 10000x magnification [17]. Then, the degree of biodegradation of the Natural Rubber Film was predicted and concluded. Tensile strength analysis was performed to observe the decreasing in tensile strength retention of the NRL films [17]. A tensile strength retention test was conducted using an Instron Universal testing machine with a crosshead speed of 500 mm/min [31].

2.4 Preparation of biodegradable glove

Glove samples was produced using laboratory dipping scales with 2 dipping tanks per scale. The first tank was filled with diluted acid, the second tank contain coagulant with the desired amount of wetting agent, the third tank contain composite latex mixture, and the fourth tank will include corn-starch. For the preparation of the samples, a lab-scale hand-forming was used. The residue and dust were removed from the former, it had been immersed in acid and then rinsed with water. After that, it has been heated to 60°C + 2°C, dipped in the coagulant solution for about 2 minutes, and then dried at 120°C for an additional 2 minutes [13]. The formers were then air-cooled to a temperature between 60 and 65 degrees Celsius, dipped for 15 seconds in the latex dispersion collected during production, and then dried at 120 degrees Celsius for 15 minutes. The last stage involves coating the moulds with cassava starch, dipping them in the 36 final tank, and allowing them to cool to room temperature. Following this procedure, latex glove samples then extracted from the mould [13].

3. Results and Discussion

The results and discussion section present data and analysis of the study including tables and figures for the result obtained.

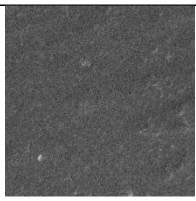
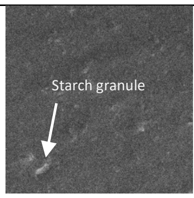
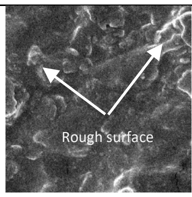
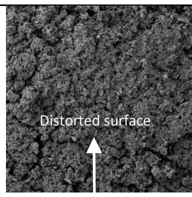
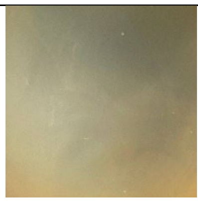
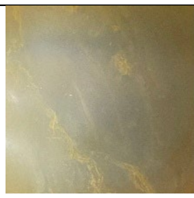
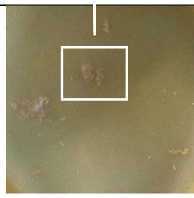
3.1 Surface morphology

Morphology Analysis of NRL- cassava-peel and soy lecithin composite films before soil burial: Table 3.1 shows the SEM image and Physical Appearance of NRL films with cassava-peel starch and soy lecithin filler loading at different concentrations ratio before biodegradation. The controlled NRL film shows a yellowish-white film with a smooth surface while NRL 8 containing high 40 phr of cassava-peel starch and soy lecithin appear to be rougher surface with white yellowish colour.

For NRL 4, the surface physically smooth from bare eye observation due to low loading of cassava peel filler in the latex. The SEM image shows a rougher surface compared to controlled NRL and the filler can be seen as white granule. The colour of soy lecithin may contribute to physical colour of film which is little bit reddish. In general, the mean particle size of the control NRL film was approximately 600 nm [25].

For NRL 5, due to the increasing filler loading for cassava-peel starch powder, the surface of film appears to be rougher compared to NRL 4. The starch particle agglomeration can be obviously seen on the film surface. NRL 8 may appear rougher surface compared to NRL 5, the cracks may be due to higher contain of fillers and the NRL cannot vulcanized well [28].

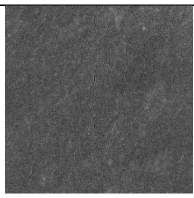
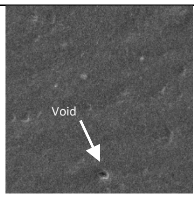
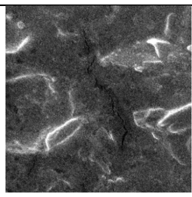
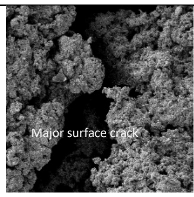
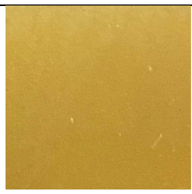
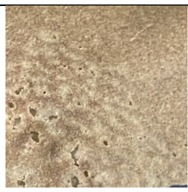
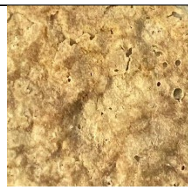
Table 3.1: The SEM images and physical appearance of NRL films before 30 days soil burial

Properties	Controlled NRL	NRL 4	NRL 5	NRL 8
SEM				
Picture				

Morphology Analysis of the NRL films after soil burial test for 30 days: After the soil burial test, there was no altering in shape and size of the samples. However, there was the formation of different degrees of biodegradation sign on the films with different cassava-peel starch loading. From Table 3.2, controlled NRL unnoticeable change was observed on the physical appearance and SEM image of the films except the sample became darker. This shows that Controlled NRL with no starch loading has undergone low biodegradation activity for 30 days as the biodegradation sign such as cracks can be observed in the film was almost negligible.

For NRL 4 with the low amount of cassava peel filler loading, not many cracks and pores were observed on the surface of the films and SEM image. The number of pores presents on the films surface increases when the amount of the cassava-peel starch loaded into the film increase as shown in NRL 5. It shows that soy lecithin may not contribute to biodegradation process. In addition, there was a large degree of degradation sign shown in NRL 8 films as many the pores and agglomeration occurred in conjunction with a large degree of discoloration of the film [21]. This phenomenon has shown that the sample had undergone a high degree of biodegradation phases with high degradation material of starch and soy lecithin filler.

Table 3.2: The SEM images and physical appearance of NRL films after 30 days of soil burial

Properties	Controlled NRL	NRL 4	NRL 5	NRL 8
SEM				
Picture				

3.2 Fourier Transform Infrared Spectroscopy Analysis

The FTIR spectra of each NRL film before soil burial test is presented in Figure 3.1 (left) while the FTIR spectra of each NRL film after soil burial test for 30 days are presented in Figure 3.1 (right). The absorption region of 600 to 800 cm is corresponding to the cis-1,4 double bonds in the polyisoprene chain [29]. The characteristic C-O-C ring vibration of the starch has caused the formation of transmittance bond at around 800-900 cm [30]. After soil burial test, the transmittance decreases gradually as the amount of starch loading increase. This shows that the degradation of starch is at a higher rate on the films with higher loading of cassava peel and soy lecithin fillers [29].

In the region of 1000 to 1200cm-1, the band becomes more prominent in the films with an intermediate amount of cassava-peel loading (NRL5&8). This region is corresponding to the CH, CH2 and CH3 bending vibration in the starch [31]. A small degree of transmittance band occurred in the region between 1500cm-1 to 1700cm-1 which represents the amide I and amide II bands of the protein molecules present in the NRL [29]. A higher density of band was observed in NRL samples with lower loading of cassava peel starch (NRL 1,2 and 4).

FTIR spectra have detected a subtle change in the transmittance band at the CH2 and CH3 stretching vibration region between 2800 cm and 3000 cm [32]. The band is predominant in the NRL film with a lower number of fillers loading especially cassava peel filler as the long-chain rubber matrix molecules are in higher composition in the films with lower starch loading. The decrease in transmittance band indicates that different degree of degradation has occurred over the entire surface of all sample. Besides, the oxidation process may be occurred and caused the side chain of the polymer to be unzipped [27].

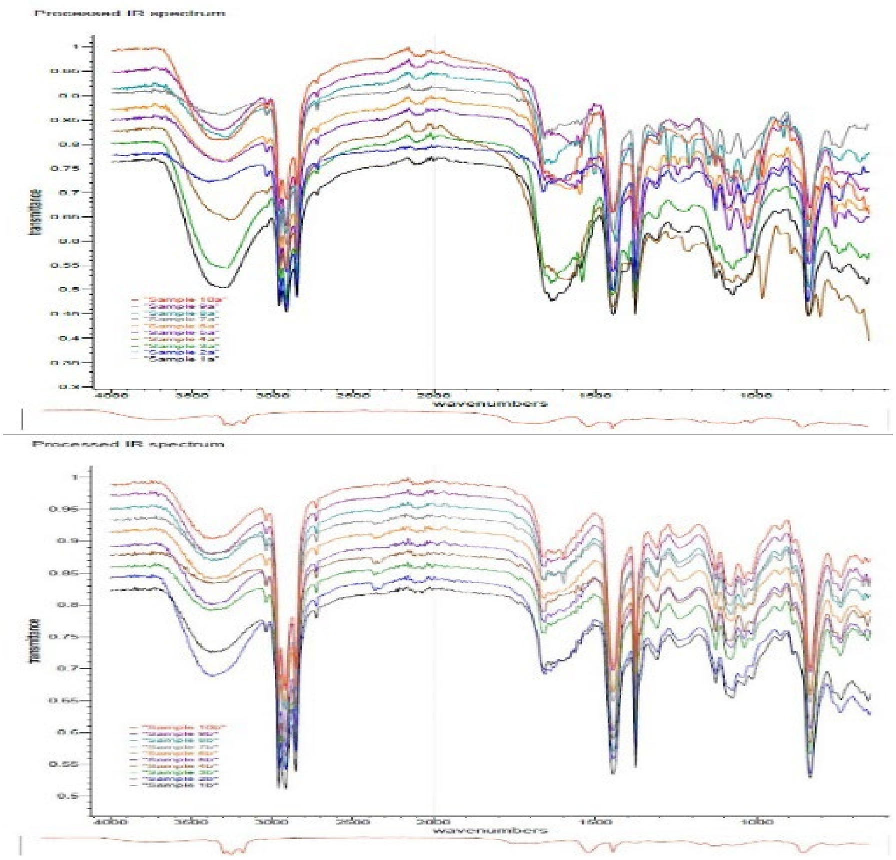


Figure 3.1: FTIR spectra of the cassava-peel starch loaded NRL films before soil burial test and after soil burial test.

3.3 Mechanical Analysis and optimisation using Taguchi

The data of stress at break and strain at break of prepared films from tensile strength testing which to be optimised were recorded as Table 3.3.

Table 3.3: Design of experiment completed table with responses

Designation	A: Cassava peel loading filler(phr)	B: Soy lecithin filler loading (phr)	C: Mixing speed (rpm)	Stress at break (MPa) (Response 1)	Strain at break (%) (Response 2)
Control	0	0	350	0.041	601.42
A2B1C1	40	30	350	0.049	616.245
A0B0C2	20	20	450	0.054	697.772
A1B1C2	30	30	450	0.060	798.188
A0B2C1	20	40	350	0.054	685.965
A2B0C1	40	20	400	0.045	614.020
A1B0C1	30	20	350	0.050	656.518
A1B2C1	30	40	400	0.056	771.380
A2B2C2	40	40	450	0.043	612.490
A0B1C1	20	30	400	0.059	717.490

The sum values of the SN ratio for stress at break were calculated to be positive values for all levels and factors as shown in Table 3.4. The values then were plot as shown in Figure 3.2.

Table 3.4: Sum of SN ratios for stress at break for prepared samples

Level/Factors	Sum of SN ratio		
	A	B	C
0	-25.085	-26.070	-25.670
1	-25.068	-25.056	-25.672
2	-26.833	-25.960	-25.644

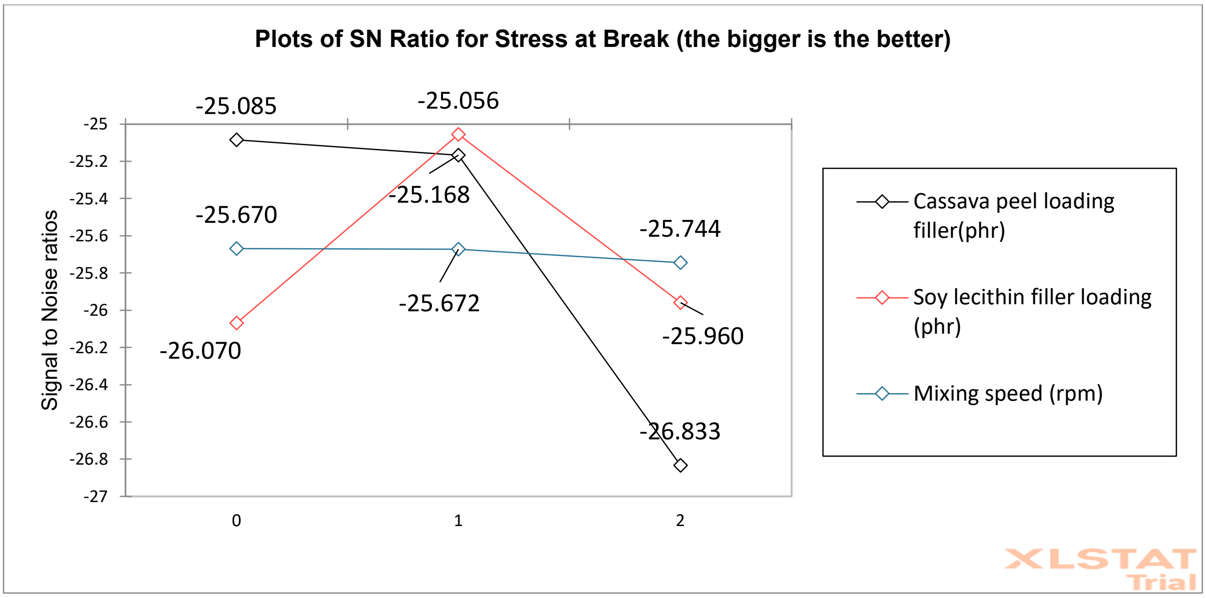


Figure 3.2: Signal Noise (SN) plot of stress at break for prepared films

Based on Figure 3.2, it was observed that the optimum condition of stress at break for three levels and three factors is at A1 (cassava peel filler at 30 phr), B1 (soy lecithin filler at 30 phr), and C2 (mixing speed at 450 rpm). Thus, the Taguchi method suggests that biodegradable glove should be prepared under condition where the cassava peel loading filler is 30 phr, soy lecithin loading of filler is 30 phr, and mixing speed is 450 rpm (A1B1C2) to obtain high stress at break. The value of stress at break for these conditions is 0.060 MPa.

The sum values of the SN ratio for stress at break were calculated to be positive values for all levels and factors as shown in Table 3.5. The values then were plot as shown in Figure 3.3.

Table 3.5: Sum of SN ratios for strain at break for prepared samples

Level/Factors	Sum of SN ratio		
	A	B	C
0	56.906	56.328	56.535
1	57.377	56.984	56.605
2	55.767	56.738	56.886

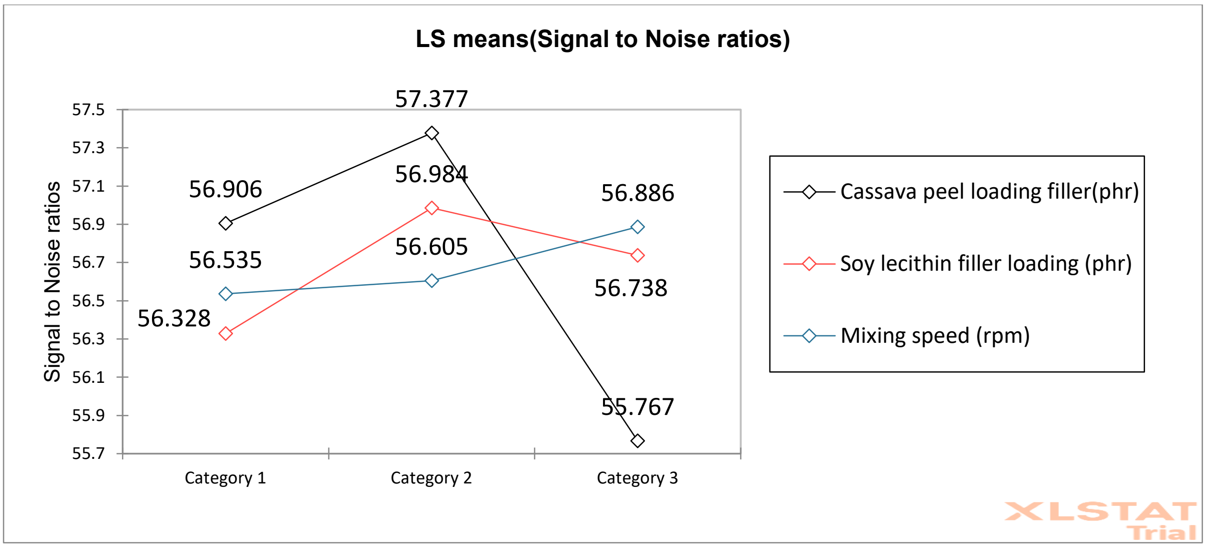


Figure 3.2: Signal Noise (SN) plot of stress at break for prepared films

From Figure 3.2, it is observed that the optimum condition of strain at break for three levels and three factors is at A1 (cassava peel loading filler is 30 phr), B1 (loading of soy lecithin filler at 30 phr), and C2 (mixing speed at 450 rpm).

The Taguchi method suggests that biodegradable glove sample shall be prepared at cassava peel starch filler loading at 30 phr, soy lecithin loading filler at 30 phr, and mixing speed at 450rpm (A1B1C2) conditions to obtain highest strain at break. Thus, from these Taguchi analyses, the conditions at which glove can be produced was determined which is A1B1C2 (NRL 3).

4. Conclusion

In the nutshell, it was shown the model of design experiment carried out using Taguchi method suggest the condition of A1B1C2 which has the highest stress at break (0.06MPa) and strain at break (798%) to be used in dipping process for biodegradable glove production. The glove produced was smooth with no cracks observed. The surface of film was observed by SEM and concluded the high volume of fillers was not suitable for glove preparation as the fillers may disturb the vulcanization thus reduced the mechanical strength significantly. The other conditions for dipping process such as temperature of vulcanization and mixing time can be further studied in detail. Finally, response surface method also can be used instead of Taguchi to get more accurate experiment design.

Acknowledgement

This research was made possible by funding from research grant number H089 provided by the Research Management Center, Universiti Tun Hussein Onn Malaysia. The authors would also like to thank the Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia for its support.

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