

# Surfactant usage in nitrile rubber film development through metal plate dipping process

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**Abstract.** Developing rubber film in the lab is important for studying the mechanical changes on gloves with different developed formulation. The process in the lab differs from the factory, as the rubber film is formed from dipping a metal plate into the compounded mixture, while the factory uses a well-crafted former to make their gloves in a controlled environment. One issue arises during preparation rubber film in laboratory is the coagulant was unable to uniformly coat the metal plate, which result in producing uneven rubber films. Thus, this work focuses on the incorporation of surfactant into the coagulant solution ( $\text{Ca}(\text{NO}_3)_2$ ) to allow a uniform coating. Two concentrations of two types of surfactants are tested by measuring the contact angle of coagulant to the metal plate. 0.5% of Wetpol 800 surfactant provides an even coating of rubber film on the metal plate after dipping through visual inspection compared to the  $\text{Ca}(\text{NO}_3)_2$  alone. The rubber film formed has an average thickness of 0.065 mm, tensile strength of 22.348 MPa, ultimate elongation 1388.4 %, modulus E100 and E300 of 1.377 and 0.731 respectively.

## 1 Introduction

Rubber is a polymer that has been widely used in the industry and is present in many consumer products like tyres, gloves, and shoes due to its elasticity and insulation properties. This polymer usually contains a long chain of carbon-carbon double bonds on its backbone, after polymerisation of conjugated dienes monomers. Rubber branches out into different types depending on the type of side groups attaching on the carbon backbone. One of it is nitrile butadiene rubber (NBR), where it contains a nitrile ( $-\text{CN}$ ) side group [1].

NBR has several advantages over natural rubber, such as having higher mechanical, chemical and thermal resistance properties, and does not contain any proteins that cause allergy to some people. In rubber factory, NBR gloves are produced through a series of steps,

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starting with cleaning the former with cleaning agents and drying. After dried, the former is dipped into a coagulant tank containing mainly calcium salt solution (calcium nitrate or calcium chloride). The coagulant allows the rubber latex to adhere and disperse on the former. To assist peeling off the glove from the former, an anti-tack dispersion, e.g. calcium carbonate, is added to the coagulant. The former with the dry coagulant film is then dipped into the compounded latex mixture and allowed to dry before curing in the oven. The gloves are lastly treated with powder slurry, chlorine, or polymer solution, making the surface less tacky [2]. In the lab, this process can be replicated in a smaller scale with metal plate as former instead. One of the problems that can occur in the process is the coagulant unable to spread evenly and shrink into droplets on the metal plate. This will make the latex concentrate at the coagulant droplets on the metal plate and develop an uneven film.

To solve this, surfactants can be added into the coagulant mixture. Surfactants are amphiphathic molecules that have dual characteristics with hydrophilic head and hydrophobic tail. It can reduce the surface tension of a substance and allow the substance to spread and wet a surface properly [3]. However, determining the type and concentration of surfactant plays a significant role of the effectiveness in spreading the coagulant on the metal plate. If the concentration of the surfactant is too low, the coagulant will still unable to spread evenly on the plate and NBR will not adhere homogenously causing the film developed to have pin-hole and have undesirable mechanical properties [4]. On the other hand, if the concentration of surfactant is too high, the coagulant will overspread, slip off the metal plate and unable to adhere on the metal plate, which is a common issue seen in agricultural surfactants used on biological surfaces [5].

In this research work, a series of coagulant 15% wt of calcium nitrate ( $\text{Ca}(\text{NO}_3)_2$ ) solution was prepared with and without the surfactants added into it. The two surfactants used are Wetpol 800, an organosilicone surfactant, and Tween 20, a polysorbate surfactant; both tested at 0.005% wt and 0.5% wt. The spreading of coagulant on the metal plate is measured with the contact angle of the coagulant on the metal plate. The coagulant solution that has the lowest contact angle will be selected. The effectiveness of adhesion of latex on the metal plate is then compared between the selected coagulant and pure  $\text{Ca}(\text{NO}_3)_2$  solution. The properties of the rubber film produced is measured: average thickness, tensile strength, ultimate elongation, modulus at 100 % and 300 % elongation (E100 and E300).

## 2 Materials and methods

### 2.1 Raw materials

NBR, zinc oxide (ZnO), zinc 2-mercaptobenzimidazole (ZMBT) and  $\text{Ca}(\text{NO}_3)_2$  were supplied by Zam Scientific & Supply Sdn Bhd. (Malaysia). Analytical grade potassium hydroxide (KOH) was purchased from System Sdn Bhd. (Malaysia). Surfactants Wetpol 800 and Tween 20 were sourced from “Best\_herbalmart” and “Future Food” respectively at the online shopping platform Shopee.

### 2.2 Preparation of compounded mixture

The compound mixture was first mixed with an RW20 digital overhead stirrer (IKA, Germany) at 250 – 280 rpm at 1 hour, according to the mass and sequence listed in Table 1. The mixture was then precured at 70 °C for 30 minutes, and stored in the fridge at 2 – 8 °C overnight. The mixture was again stirred with the overhead stirrer for another hour before used for dipping.

**Table 1.** Formulation of compounded latex mixture.

Components	Average total solid content (TSC) (%)	Dry phr
NBR	44.0757	100
Water	-	-
KOH	10	0.8
ZnO	38.6386	1
ZMBT	40.1811	1
Sulfur	47.8399	1

### 2.3 Preparation of coagulant solution

The coagulant solutions were made according to the formulations below in 100 mL volumetric flask each.

**Table 2.** Formulation of coagulant solution.

Coagulant solution	Formulation
A	15 % Ca(NO <sub>3</sub> ) <sub>2</sub>
B	15 % Ca(NO <sub>3</sub> ) <sub>2</sub> + 0.005 % Wetpol 800
C	15 % Ca(NO <sub>3</sub> ) <sub>2</sub> + 0.005 % Tween 20
D	15 % Ca(NO <sub>3</sub> ) <sub>2</sub> + 0.5 % Wetpol 800
E	15 % Ca(NO <sub>3</sub> ) <sub>2</sub> + 0.5 % Tween 20

### 2.4 Contact angle measurement

A drop of each coagulant solution was dropped on the middle of the steel plate. A picture was taken with Samsung Galaxy A34 5G phone at 10x magnification. The contact angle of the drop was measured using the drop analysis plugins from the ImageJ software. The coagulant solution that has the smallest contact angle will be selected for the dipping process.

### 2.5 Dipping process

13 cm x 15.5 cm steel plates were first heated at 70 °C in the oven, dipped into the selected coagulant solution for 2 minutes and dried back at 70 °C for 15 minutes. The steel plates were dipped back into the compounded mixture for 7 minutes followed by gentle leaching in water and vulcanised at 100 °C for 30 minutes in the oven. The process is repeated for all coagulant formulations.

### 2.6 Mechanical properties

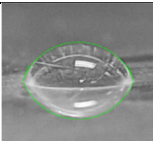
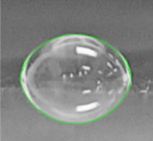
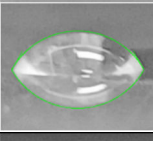
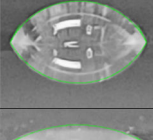
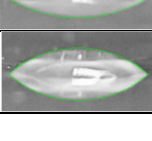

The thickness for the rubber film produced is measured with an electronic micrometre. The tensile test and ultimate elongation of ten rubber film samples were measured with a Tinius Olsen Universal Testing Machine (Tinius Olsen, USA) with 500 mm/min as the cross-head speed. Tensile strength, modulus at 100% and 300% elongation (E100 and E300) of the tested samples recorded. The procedure followed the ASTM D412-16 standard [6]. The mechanical properties of the rubber film samples are examined under ASTM D6319 standard.

## 3 Results and discussion

### 3.1 Contact angle

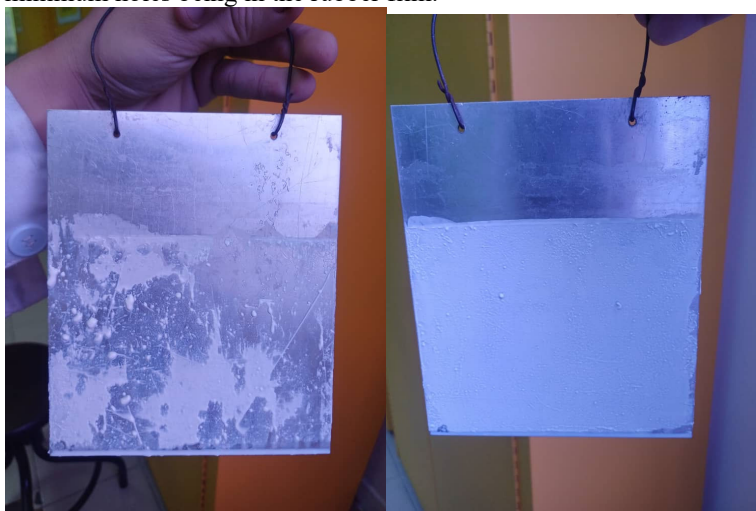
The contact angles of deionised water and each coagulant solutions are listed in Table 3. The  $\text{Ca}_2(\text{NO}_3)_2$  solution (A) has a higher contact angle than pure deionised water. The water molecules attract calcium and chloride ions and cause the water molecules to aggregate together [7]. This causes the contact angle of a salt solution to be higher than water, making the coagulant to shrink into droplets rather than spread evenly on the metal plate. All the formulation B – E that contain surfactants have lower contact angle than pure water and salt solution. Surfactants can reduce the liquid-vapour interfacial tension and the solid-liquid interfacial tension, which effectively reduce the contact angle between the water droplets and the metal surface. That is why an extremely low concentration of surfactant used in B and C can reduce the contact angle lower than pure deionised water, and a higher concentration of surfactants causes the contact angle to further reduce for better adhesion and wettability of the coagulant on the metal plate. The type of surfactants used will also affect the interfacial tension and contact angle. Wetpol 800 is a non-ionic silicone surfactant, a type well-known for its ability to greatly decrease the surface tension of an aqueous solution upon a surface. It is widely used in fertiliser and pesticide mixture for foliar application, allowing the agrochemicals to wet and adhere on the surface much longer [8]. Tween 20 is a non-ionic polysorbate surfactant that is used in stabilising oil-water emulsion present in food and cosmetic products, preventing the oil and water to separate out. Literature review from previous studies showed that for a surface to be superhydrophilic, where the aqueous solution can wet the surface well, the contact angle will need to be below  $40^\circ - 50^\circ$  [9-11]. Therefore, coagulant solution D is selected for the dipping process, as it has the lowest contact angle (smaller than  $40^\circ$ ) and spread evenly on the plate.

**Table 3.** Contact angle of coagulant solutions.

Coagulant solution	Water droplet		Contact angle ( $^\circ$ )
Deionised water			69.757
A			77.094
B			60.627
C			62.081
D			32.531
E			41.263

### 3.2 Physical observation

Figure 1 shows the rubber coating on the metal plate after the dipping process. The metal plate that was dipped into coagulant solution A has a poorer rubber film coverage as predicted from its high contact angle. Since coagulant solution A tends to shrink into droplets and forms concentrated spots on the metal plate after drying. The rubber will stick on these concentrated spots and not wet the entire metal plate. Meanwhile, coagulant solution D, having the lowest contact angle, can produce an even rubber coating on the metal plate, since it is able to wet and spread evenly on the metal plate, allowing the rubber to also coat evenly on the metal plate with minimum holes being in the rubber film.



**Fig. 1.** Rubber coating on metal plate with coagulant solution A (left) and D (right).

### 3.3 Mechanical properties

Table 4 displays the properties of the rubber film produced from coagulant solution D and the minimum requirement of rubber for medical nitrile gloves according to ASTM D6319. From the table, it shows that the introduction of surfactants in coagulant solution can produce rubber films that satisfy the minimum requirement of the ASTM D6319 standard [12]. Further researchers can reliably produce uniform rubber films that has similar properties than the ones produced from factory. This allows researchers to produce and characterise rubber films that have different formulations from what produced from industry while following ASTM D6319 standard.

**Table 4** Properties of rubber film produced.

Properties	Minimum requirement	Values
Thickness (mm)	0.05	0.065 ± 0.006
Tensile strength (MPa)	14	22.35 ± 4.26
Ultimate elongation (%)	500	1388.4 ± 82.5
E100 (MPa)	-	1.377 ± 0.165
E300 (MPa)	-	2.194 ± 0.241

## 4 Conclusion

The wettability of pure  $\text{Ca}_2(\text{NO}_3)_2$  solution and coagulant solutions with 0.005 % and 0.5 % of Wetpol 800 and Tween 20 evaluated through the contact angle measurement. The coagulant solution with 0.5 % Wetpol 800 selected for dipping as it has the lowest contact angle of  $32.531^\circ$ . This coagulant solution can produce uniform rubber films with average thickness of 0.065 mm, tensile strength of 22.35 MPa, ultimate elongation 1388.4 %, modulus E100 and E300 of 1.377 and 2.194 respectively, which satisfied the minimum requirement of nitrile medical gloves following the ASTM D6319 standard.

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