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Research article

The Impact on Properties of Block Rubber of Using Adulterated Coagulants in Cup Lumps of Natural Rubber

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Abstract: Cup lumps are raw materials used in block rubber production and commonly contain adulterants among the chemicals used for rubber coagulation. This study aimed to identify the composition and concentration of components in commercial coagulants for producing cup lumps in the Northeastern and Northern regions of Thailand. A further goal was to study the impact of using these cup lumps to make block rubber. Detection of coagulant concentration by ion chromatography found that 25% of the samples were composed of formate ions and 50% of the samples were composed of sulfate ions ranging from 17.89 to 93.46% and 36.17 to 97.74% by weight, respectively. Among the adulterated coagulants, they were mixed with formate ions and chloride ions in 18.75% of the samples and sulfate ions mixed with chloride ions in 6.25% of the samples. Additionally, block rubber containing adulterants had Po, PRI, and Mooney viscosity values that meet the lower grade STR 20 standard. Block rubber made from cup lumps coagulated using formic acid showed physical properties that met the STR 5 standard.

Keywords: Adulterated coagulant; Block rubber; Cup lumps

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1. Introduction

Cup lump natural rubber is the starting raw material in block and crepe rubber production. It has a production volume of 1.49 metric million or 32% of Thailand total production of raw rubber materials [1]. Most of this is produced in the northeastern region accounting for 80% of the country's output. Cup lumps of rubber are obtained from latex coagulated in a cup. The shape of the cup lumps is, therefore, that of its container. They have many sizes, both small and large. The volume and weight of cup lumps are unspecified, ranging from 100-500g [2]. Cup lumps are produced using acid as a coagulant or allow natural coagulation with no acid. Non-acidic cup lumps of natural rubber do not settle properly. Over an extended period, this will cause spoilage, air bubbles and porosity in the cup lumps. The result will be an initial weakness and low viscosity in addition to having poorer physical properties than if acid was used to coagulate latex [3].

Additionally, malodors will be generated due to the volatile organic compounds in non-rubber, including ketones, aldehydes, aromatic compounds, esters, acids, alkanes, alcohols and cyclic compounds [4-5]. Decomposition results in the foul 'rotten egg' smell of hydrogen sulfide and mercaptan, while acid coagulation of rubber effectively separates the non-rubber from the rubber. More protein is in the serum fraction than in the rubber, resulting in a less foul odour. Only good quality cup lumps can produce good quality block rubber [6].

The Rubber Research Institute of Thailand recommends using formic acid as a coagulating agent as it is a readily degradable organic acid with no residue in the product [3, 7]. However, some manufacturers of rubber coagulating agents mixed various adulterants into their products and some producers added other materials to the coagulants. In 2021, the ROAT received a letter from the Thai Rubber Association (TRA) complaining about using adulterated latex coagulants to make cup lumps, especially in Thailand's North and Northeast regions. In particular, there is a problem of degrading cup lumps with ammonia when the rubber is cut into small squares inside the cups. This is done regardless of the damaged product. The practise is widespread in the upper northeastern provinces of Bueng Kan, Nong Khai and Udon Thani. Additionally, formalin was added to latex to maintain a new condition before delivery to latex processors who make concentrated latex or rubber sheets. Other contaminants included vulcanized rubber and remnants of material from manufacturing foam rubber. Additionally, soil, sand, cement, molasses and inappropriate binders such as sulfuric acid and chloride salts were found in the cup lumps. In 2013, the Rubber Research Institute of Thailand also received complaints from the TRA that cup lumps produced in the northeastern region were not of good quality. As a result, tire manufacturers did not accept rubber shipments from the northeast region due to residual sulfuric acid in the products. Sulfuric acid causes the rubber to become sticky and dark in colour. This affects adhesion between the wire and the rubber of tires. There is also a problem with the joints of the tire valves. They become disconnected from the rubber during tire assembly. Moreover, exposure to sulfuric acid can irritate the skin, eyes, and nose. The formation of gaseous sulfur compounds such as SO_2 and H_2S can also poison the environment. In light of these problems, Tassanakul [3] prepared a manual for producing good quality cup lumps of natural rubber for farmers to implement. This manual also covers good manufacturing practices (GMP) for making crepe rubber from high-quality cup lumps. Such information has been developed as a good manufacturing practice for crepe rubber production [8-9], a good agricultural practice for rubber to produce fresh latex [10], and a good agricultural practice for rubber to make cup lumps [11]. These are optional rubber standards that promote and support the dissemination of quality control of agricultural products.

The objective of the current study was to determine the composition of each commercial adulterated coagulant in the northeastern and northern regions of Thailand. We studied the chemical and physical properties of block rubber that contain adulterants used in making cup lumps of natural rubber. Additionally, procedures are proposed to prevent the use of adulterated coagulants in producing cup lumps. This very serious problem causes great damage to Thailand and the world rubber industry. Overall, the impact derived from the scientific finding of using commercial adulterated coagulants must be solved by the relevant organization.

2. Materials and Methods

This research was a quantitative study. It examined 60 rubber thickening agents found in the northeastern and northern regions. The experiments were divided into three phases as follows. In the first step, 50 of 60 coagulating agents were selected by random sampling. This was done with a quick test kit that detected the presence of organic acids using Gentian violet [12]. The samples were checked for sulfate adulteration using a 10% barium chloride solution to adopt the ISO 2480:1972 [13]. Then the samples were checked for chloride salts adulteration with a 10% silver sulphate solution in a process for adopting the ISO 2479:1972 [14]. The pH values of the samples were checked using litmus paper.

In the second step, 16 of 60 samples were selected for analyzing the concentrations of formate ($HCOO^-$), sulfate (SO_4^{2-}) and chloride (Cl^-) ion by using ion chromatography.

For the third step, natural rubber latex was subjected to five (5) types of rubber coagulates. They were (1) formic acid, (2) sulfuric acid, (3) calcium chloride salts, (4) formic acid with calcium chloride salts, and (5) sulfuric acid with calcium chloride salts. All of them are commercial grade. Label directions were followed for each coagulant to form cup lumps. This produced crepe rubber that was sent to a shredder and then to a drier in the block rubber factory at the Factory Management Division 4, Rubber Authority of Thailand, Khun Han District, Sisaket Province. To study the chemical and physical properties of these materials, including the amounts of impurities (dirt), the ash content (ash), volatile matter content (V.M.), nitrogen content as well as the initial plasticity (Po), plasticity retention index (PRI) and Mooney viscosity (Vr) were compared to Standards STR 5, STR 10 and STR 20 [15]. This study was conducted in 2020-2021 at the Factory Management Division 4, Khun Han District, Sisaket Province. Additionally, a small group meeting was held to determine measures to prevent the use of adulterate coagulants in the production of cup lumps and improve the quality of rubber.

3. Results and Discussion

3.1 Determination of the presence of formic acid, sulfuric acid, chloride salts of latex coagulants using a test kit and pH using litmus paper

A total of 50 coagulants were sampled. They were solutions packaged in plastic and glass bottles as well as large plastic containers. Package volumes ranged from 750 mL to 5 L. It was found that some brands of latex coagulants had the same logo, but their colours were different. Some were colourless and transparent, while other solutions were light yellow, dark yellow and black. Some solutions of the same brand had different colours. There were coagulants of the same brand for making cup lumps and rubber sheets. Some labels specified the methods of use, while others did not. Some could be used directly, while others required dilution. None of the latex coagulants specified its type and concentration. The date of manufacture was not determined, nor was the expiry date, except for one sample that contained formic acid, identified as "Formic". That one sample had detailed information clearly stated on its label. It accounted for 2% of all samples tested. Gentian violet solution was used to determine the type of organic acids present, for the organic solution will appear blue-violet. If the solution is inorganic, it will be changed from blue-violet to green or yellow, depending on the acidity of the solution. The yellow indicates the pH of 1–3, while the green colour has a pH range of> 3-5.

It was found that only 11 samples (22%) contained only organic acid as formic acid. A test for sulfuric acid was done using a barium chloride solution. It was observed that 17 samples (34%) caused the solution to change from clear to opaque as barium sulfate precipitation. A test for chloride salts s was done using a silver sulphate solution. Silver chloride was precipitated as a discolouration of the clear solution to one that was milky-white. Five (5) samples (10%) were found to contain organic acids (formic) mixed with chloride salts. Mixtures of sulfuric acid and chloride salts were detected in 17 samples (34%), as shown in Table 1.

Table 1. The results of formic acid, sulfuric acid and chloride salts presence in various coagulants

Type of Coagulants	Number of samples	%
1. Formic acid	11	22
2. Sulfuric acid	17	34
3. Chloride salts mixed with formic acid	5	10
4. Chloride salts mixed with sulfuric acid	17	34
Total	50	100

Most (88%) of the coagulants had a pH of 1, which is strongly acidic. It was found that most (78%) of the coagulating agents were adulterated using both inorganic acids along with sulfate or chloride salts. This is consistent with the study of Tassanakul [16], who found that commercially available coagulants were more likely to be adulterated with inorganic acids. This is because many coagulants are produced by entrepreneurs who do not regard the adverse effects on rubber producers and the environment. People have reported a burning pain on their skin after handling these products caused by vapour behaviour from sulfuric acid. Additionally, the cambium of rubber trees is damaged and darkened by a strong acid as sulfate changes to sulfide. This is consistent with other studies by Srihamongkul and Kasikanan [17]. They found that the pH of the aqueous serum obtained from coagulation is harmful to the skin and rubber surfaces in the long term. Products that contain adulterants are a fraction of the cost of those made with formic acid. There is a faster coagulating time, but the dry cup lumps are dark and sticky, while the resulting rubber is hard, lacks flexibility and has a lower viscosity.

3.2 Quantitative determination of formate, sulfate and chloride ions using ion chromatography

Sixteen (16) specific rubber coagulant samples were taken for the quantitative determination of formate ($HCOO^-$), sulfate (SO_4^{2-}) and chloride (Cl^-) ion concentrations for analysis using ion chromatography. Four (4) samples (25%) contained only formic acid. Of these, only one formic acid brand, BASF, with a formate ion concentration of 93.46%, was correctly labelled (94%). In other samples, the concentrations of ionic formate were 17.89, 31.99 and 54.35% by weight, respectively, for Dao-D, Dee-F and Reua-B brands. Eight (8) samples (50%) contained sulfuric acid as a rubber coagulating agent and the sulfate concentration was between 36.17 and 97.74% by weight. Additionally, three (3) samples (18.75%) samples contained rubber coagulating agents comprising a mixture of formate and chloride ions. The remaining sample (6.25%) was adulterated with a combination of sulfate and chloride ions, marketed as the "good quality brand, C.H.". Among the samples, two had chloride salts concentrations ranging from 30.78 and 36.78% by weight. The Rod-K brand contained formic acid, while the Hi-S brand contained only chloride salts (Table 2). A coagulant producer should show the product composition and concentration of acid on the label. This is so that farmers can use such information to make purchasing decisions. However, producers use similar brand names, which confuses the market. For example, Su-S-A and Su-S-B have similar names but differ only in colour. Those producing latex coagulants should consider the impacts on the quality of rubber, the people who produce rubber and the environment.

Table 2. Concentrations of formate ($HCOO^-$) sulfate (SO_4^{2-}) and chloride ions (Cl^-) in coagulants were determined by using ion chromatography

No.	Trade name	Concentration (% by weight)		
		$HCOO^-$	SO_4^{2-}	Cl^-
1	Rod-K	11.54	n.d	30.78
2	Ruea-B	54.35	n.d	n.d
3	BASF	93.46	n.d	n.d
4	Su-S-A	n.d	43.13	n.d
5	Su-S-B	n.d	47.88	n.d
6	Ton-Y	n.d	36.17	n.d
7	CH	n.d	40.96	0.95
8	Nak-k-l	0.68	n.d	2.50
9	Nak-k-S	0.63	n.d	2.38
10	Sing-MT	n.d	92.43	n.d
11	Phae-D	n.d	97.74	n.d
12	Suea-KC	n.d	37.64	n.d
13	Suea	n.d	53.53	n.d
14	Dao-D	17.89	n.d	n.d
15	Dee-F	31.99	n.d	n.d
16	Hi-S	n.d	n.d	36.78

3.3 Chemical and physical properties of block rubber made from cup lumps using various coagulating agents

The chemical and physical properties of block rubber made from cup lumps was studied using five (5) coagulants. They consisted of 1) formic acid, 2) sulfuric acid, 3) calcium chloride salts, 4) formic acid combined with calcium chloride salts, and 5) sulfuric acid combined with chloride salts. The formic acid concentration in sample 1 was 0.6% on a dry rubber weight basis. Samples 2-5 were used according to information on their package labels. It was found that using formic acid alone when production was controlled according to good agricultural practice (GAP) standard [11], block rubber properties showed higher Po and PRI values than from samples of cup lump rubber coagulated with sulfuric acid and chloride salts. The rubber coagulated with the latter two agents had a higher moisture content than those made with formic acid. This is because sulfates and salts absorb moisture from the air. Rubber coagulated with calcium chloride salts has a Po value of less than 30, which does not meet the STR 20 standard. This is a criterion for the lowest quality of block rubber. Its PRI value is lower than that using a formic acid coagulant. This agrees with the work of Pimrat, Poonsawat and Sahagaro [18]. It was found that using sulfuric acid above the specified level resulted in decreased PRI values and lower viscosity.

Using chloride salts causes 50% more rubber deterioration than using formic acid alone (Table 3). This is in agreement with the study of Tassanakul [19] that the use of calcium chloride alone was found to have a severe effect on cup lump quality. It makes the resulting cup lumps harder and increases its viscosity. Products made from such rubber deteriorate more quickly. The cup lump surfaces are dark and sticky. Many block rubber manufacturers have encountered problems with poor quality rubber and non-uniform properties. This affects the use of block rubber and limits the products it can use, especially in vehicle tires. Additionally, a foul smell arises from the serum water at the collection points. This affects the community and the environment in the long run [20]. When cup lumps are produced without quality control, their properties are inconsistent, especially the Mooney viscosity. This is because the natural rubber's structure comes from forming protein chains with lipids at the end of the chains, making them longer. The result increases with time, causing higher viscosity and storage hardening [21]. Xu Chen et al. [22] found different coagulation methods could also affect the storage behaviours of natural rubber since coagulation methods could affect the content of non-rubber components.

Table 3. The chemical and physical properties of block rubber using various coagulants

Type of coagulants/Standard	Dirt, max (%)	Ash, max (%)	Nitrogen, max (%)	VM, max (%)	Po, min	PRI, min	Vr ML(1+4 100 °C)
STR* 20	0.16	0.80	0.60	0.80	30.0	40.0	NS
STR 10	0.08	0.60	0.60	0.80	30.0	50.0	NS
STR 5	0.04	0.60	0.60	0.80	30.0	60.0	NS
Formic	0.011	0.32	0.34	0.40	44.7	82.0	80.7
Sulfuric	0.023	0.34	0.39	0.50	39.0	72.6	77.9
Calcium chloride	0.013	0.87	0.36	1.03	23.5	41.1	55.6
Formic + Calcium chloride	0.025	0.89	0.35	0.66	20.5	24.4	49.2
Sulfuric + Calcium chloride	0.028	0.37	0.45	0.49	29.5	69.5	61.5

Remark: STR means Standard of Thai Rubber

3.4 Recommendations for measures to control the use of non-formic acid coagulation agents in the production of cup lumps

According to the Rubber Authority of Thailand, formic acid is recommended as a latex coagulant in the production of cup lumps because it is an organic acid that readily decomposes and does not affect the environment. The cup lumps from this method have good physical properties, are safe for operators, and do not injure the cambium of the rubber trees. The surface of rubber trees does not show dryness syndrome. Manufacturers and distributors of rubber coagulation agents often mix sulfuric acid and chloride salts into their products. This affects the quality of the block rubber used in many rubber products, especially vehicle tires, which use the highest volume of block rubber. One way to control this is to enable farmers to produce good quality cup lumps. Therefore, we propose seven (7) measures to prevent rubber coagulation agents that are not formic acid, as follows:

1. Make the use of non-formic acid, or inorganic acid should be subject to regulation. Short-, medium-and long-term plans are urgently needed to develop the Rubber Authority of Thailand's policy to prohibit non-formic acid coagulants.

2. It is proposed that the Rubber Authority of Thailand confer with their legal department to formulate a Letter of Consumer Protection that will be sent to the Consumer Protection Board, which regulates advertising, labelling and contracting. The letter should ask that latex coagulants become controlled products under Section 30 of the Consumer Protection Act of 1979. If the Consumer Protection Committee on Advertising, Labeling and Contracts determines that regulation should exercise control, it will be issued under Section 31. It will regulate the production of all rubber coagulants. Such products must have a quality certificate indicating the name, place of manufacture, composition, coagulant concentration, quantity, method of use, dates of manufacture and expiry. These should be clear and attached to the container.

3. It is proposed that the Rubber Authority of Thailand begin discussions with the Department of Agriculture on this matter to address the problem of offences committed under the Consumer Protection Act of 1979 or other regulations that fall under the authority and responsibility of the Department of Agriculture.

4. It is proposed that the Rubber Authority of Thailand work with the Thai Rubber Association and request cooperation from STR 10 and STR 20 block rubber manufacturers to purchase quality rubber materials that meet the standard qualifications. Farmers should be trained to use coagulants that contain formic acid or an organic acid that produces rubber with comparable properties. There should be no adulterants such as bark, vulcanized rubber, calcium salt, bio-fermented fluids, wood vinegar, sulfuric acid or acids that claim the trade names of "organic acids", "biological acids" etc.

5. The Rubber Authority of Thailand should expedite public relations, release information on electronic media, radio, television, and newspapers, and mail promotional materials to inform governmental and non-governmental agencies concerned with this matter. The private sector, farmer groups, and rubber tree farmers should be included. The effects of non-formic acid latex coagulants should be urgently and continuously presented. This is to prevent damage to the quality of Thai rubber.

6. It is proposed that the Rubber Authority of Thailand and related agencies promote, support and motivate farmers and farmer groups to produce good quality cup lumps that meet GAP standards. This is to produce quality rubber, increase productivity and improve safety. It is a worthwhile investment to produce rubber that can be made into premium-grade crepe rubber. This will upgrade production throughout the manufacturing chain and move the industry toward sustainability and internationalization.

7. It is proposed that the Rubber Authority of Thailand modify its post-harvest and harvest management policies for rubber production according to GAP standards. The policy must be implemented to drive improved production of quality rubber to achieve maximal effectiveness.

4. Conclusions

Thailand is the world's largest producer of block rubber. This is the reason for the promotion of producing rubber in the northeastern and northern regions to meet the demands of the world market. Cup lumps are products that are popular among farmers. So, the producers of coagulants see this as an opportunity to make adulterated coagulants with sulfuric acid and chloride salts. This is widespread in the industry and it is hard to control. Studies have shown that coagulants are sold that contain inorganic rather than organic acids. In the production of block rubber, manufacturers cannot distinguish which coagulants have been used in the cup lumps. Additionally, rapid detection of sulfate adulteration with a barium chloride solution and checking for adulteration with chloride salts s using a silver sulphate solution can be done. If it is desirable to determine the concentration of various adulterants, ion chromatography can determine the concentrations of these substances. The results showed that the number of samples containing sulfuric acid was more than those with formic acid. Coagulants were also adulterated with formic acid mixed with chloride salts and sulfuric acid mixed with chloride salts s. According to the GAP, using formic acid in cup lumps to produce block rubber showed that the chemical and physical properties pass the STR 5 standard for block rubber. Other block rubbers were adulterated with sulfate and chloride salts. Their flexibility deteriorated with viscosity so that it was lower than the STR 20 standard, which was Thailand's lowest grade of block rubber. Therefore, the Rubber Authority of Thailand, as a central organization and management of the entire system, should be strictly implemented for using adulterated coagulants not to have a broad impact on the properties of block rubber.

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