Rubber Recovery from Waste Latex Sludge Using Sulfuric Acid

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Abstract – Waste latex sludge, which is null by-product of concentrated latex manufacturing, was digested to retrieve the rubber by using sulfuric acid. It was found that the acid concentration affected on the amount and purity of retrieved rubber. Sulfuric acid at concentration more than 10% by weight completely digested waste latex sludge and gave rubber 10% by weight. The quality of retrieved rubber were examined for Mooney viscosity (MV), plasticity retention index (PRI), nitrogen content, and ash contents. The residue solution was evaporated and crystallized. The crystal structure was determined using power X-ray diffractometer.

Keyword: natural rubber, waste latex sludge, sulfuric acid, concentrated latex, rubber recovery
1. Introduction

The concentrated natural rubber latex (NRL) is always produced by using centrifugation of fresh field NRL. NRL is not only consisted of NR particles but also non-rubber parts such as magnesium (Mg). The NRL contains high Mg has an impact to the centrifugation process and the quality of concentrated latex. Therefore, it is necessary to remove the magnesium from NRL. The traditional method is adding of diammonium hydrogen phosphate (DAHP) to NRL and leave for more than 12 hours. The reaction of DAHP with Mg resulting in solid sludge formation and precipitation. The solid sludge characteristics have been investigated and reported their advantages for agronomic relevance since the year 2000 [1,2]. Basic composition of this materials are rubber hydrocarbon, nitrogen, magnesium, and phosphorus for 12.5%, 3.3%, 12.2%, and 14.7% by wt. (dry weight), respectively [3]. Eventhough these material can be comparable used as commercial phosphatic fertilizers but the limitations, heavy metal content, and unsuitability to certain soils and crops must be concerned as well as the effect of rubber hydrocarbon which is not easily decomposed in soils.

This is the first report deals with the rubber recovery from waste solid sludge and occurrence of high purity ammonium magnesium sulfate.

2. Experimental

The sludge, collected form latex centrifuge machine, was supplied by Chalong Concentrated Latex Co., Ltd. and was used immediately. The sludge was then digested with sulfuric acid solution by varying acid concentration at room temperature as illustrated in figure 1.

The sulfuric acid for 5% by wt. was not strong enough to digest the waste sludge while the higher concentrations of sulfuric acid digest the solid sludge result in floating retrieved rubber because the rubber density is lower than 1.

The retrieved rubber was separated, washed with water, sheeted, and dried in air oven at 70°C. The composition of rubber was analyzed using Thermo gravimetric analyzer (TGA). The rubber structure and molecular weight were characterized using Fourier transform infrared spectroscopy (FT-IR) and Gel permeation chromatography (GPC), respectively. Quality of the retrieved rubber was compared with standard NR which is classified in STR5L grade.

The residue waste water was characterized and was crystallized at room temperature. The crystal structure was characterized by using X-ray Diffractionmeter (X’Pert MPD, Philips, Netherlands).

3. Results and Discussion

The compositions of waste latex sludge and retrieved rubber from TGA is illustrated in figure 2. The waste latex sludge composed with moisture, rubber hydrocarbon and inorganic substance for 35%, 20%, and 45% by wt., respectively. The retrieved rubber by digestion of waste latex sludge with sulfuric acid composed with rubber for more than 95% by wt. Most of inorganic substance were removed and dissolved in the solution of sulfuric acid.

![Figure 2 TGA thermogram of solid sludge before and after digestion with sulfuric acid.](image)

![Figure 3 FT-IR spectra of retrieved rubber and standard NR (STR5L).](image)
The FT-IR spectra of the retrieved rubber compared with standard NR (STR5L) is shown in figure 3. The major characteristic peaks of standard NR are at 2962 cm\(^{-1}\) (CH\(_3\) asymmetric stretching), 2855 cm\(^{-1}\) (CH\(_2\) asymmetric stretching), 1650 cm\(^{-1}\) (C=C stretching), 889 cm\(^{-1}\) (CH\(_3\) wagging), and 837 cm\(^{-1}\) (=C-H wagging). [9] For the retrieved rubber, the occurrence of peak at 713 cm\(^{-1}\) (O-H out-of-plane bend) and the broader peak at 3340 cm\(^{-1}\) (hydroxyl group, H-bonded OH stretch) indicating the higher number of water molecules surrounding the membrane and functional ended group of the retrieved rubber might be hydroxyl and carbonyl.

The GPC result of the retrieved rubber as shown in figure 4 was found that the average molecular weight by number (M\(_n\)) and the average molecular weight by weight (M\(_w\)) of the retrieve rubber are 90,000 and 313,000 g/mol, respectively. The molecular weight, represented rubber molecule chain length, of the recovered rubber from sludge is very low compared with the standard NR (M\(_n\) = 442,400 g/mol, M\(_w\) = 1,745,500 g/mol).

The rubber properties of retrieved rubber compared with standard NR (STR5L) is illustrated in table 1. The Mooney viscosity and initial plasticity of the retrieved rubber are lower than that of standard NR due to the low molecular weight of retrieved rubber but the PRI of retrieved rubber is higher than that of standard NR indicated that the retrieved rubber is not easily oxidation degraded because of some natural antioxidant contamination. The results of this study hold promise as an apparent feasible solution to the problem of waste disposal in the NR latex concentrated factories with cost effectiveness and gains the valued NR from waste sludge which is cheap throw away material.

The waste water from sludge digestion with 10% by wt. sulfuric acid is dark brown color with pH = 5.8 and was analyzed for BOD and COD. The results was found that the BOD and COD were 2200 and 27200 mg/L, respectively. The residue digestive was concentrated by evaporation at 100°C and allowed the crystals formation at room temperature.

The occurrence crystals are white to clear color and the gloss is vitreous as shown in figure 4. The powder X-ray diffraction pattern of the crystals as shown in figure 5 were in good agreement with Boussingaultite mineral (RRUFF ID- R070597.9)\([6,7]\) and the refine unit cell parameters are \(a=9.327(3)\) Å, \(b=12.600(4)\) Å, \(c=6.210(1)\) Å, \(b=107.091(6)\)°, and \(V=697.6(8)\) Å\(^3\).

### Table 1 Properties of retrieved rubber from solid sludge

<table>
<thead>
<tr>
<th>Properties</th>
<th>Rubber</th>
</tr>
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<tbody>
<tr>
<td>Mooney Viscosity [ML1+4, 100°C]</td>
<td>60, 49</td>
</tr>
<tr>
<td>Initial Plasticity (Po)</td>
<td>40, 33</td>
</tr>
<tr>
<td>Plasticity Retention Index (PRI)</td>
<td>77, 93</td>
</tr>
<tr>
<td>Nitrogen content (%)</td>
<td>0.3, 0.9</td>
</tr>
<tr>
<td>Ash content (%)</td>
<td>0.1, 3.5</td>
</tr>
</tbody>
</table>

The rubber removal from waste sludge using sulfuric acid is feasible solution to solve the problem of waste disposal in the NR latex concentrated factories in any developing country like Thailand. It is not only to retrieve high value NR but also the magnesium ammonium sulfate, which is essential nutrient in crop production, from null throw away materials.

### 4. Conclusions

The rubber removal from waste sludge using sulfuric acid is feasible solution to solve the problem of waste disposal in the NR latex concentrated factories in any developing country like Thailand. It is not only to retrieve high value NR but also the magnesium ammonium sulfate, which is essential nutrient in crop production, from null throw away materials.
5. Acknowledgement

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References


