

Analysis Method

Simple test kit based on colorimetry for quantification of magnesium content in natural rubber latex by miniaturized complexometric titration without using masking agent



Nutthaporn Malahom^a, Purim Jarujamrus^{a,*}, Rattapol Meelapsom^a,
Atitaya Siripinyanond^b, Maliwan Amatotongchai^a, Sanoe Chairam^a

^a Department of Chemistry and Center of Excellent for Innovation in Chemistry, Faculty of Science, Ubon Ratchathani University, Ubon Ratchathani 34190, Thailand

^b Department of Chemistry and Center of Excellent for Innovation in Chemistry, Faculty of Science, Mahidol University, Rama VI Road, Bangkok 10400, Thailand

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ABSTRACT

A simple, low-cost and portable field test kit based on colorimetry with detection by naked eye was developed for determination of magnesium content in natural rubber latex (NRL). The miniaturized complexometric titration between Mg²⁺ and EDTA without any masking agent was a key reaction in this development, which was designed according to the concept of green chemistry by reduction of waste generation and chemical and time consumption. The system enabled quantification of magnesium content in NRL at low concentration with the detection limit being <50 mg L⁻¹, small sample volume uptake (0.18 g, sampling by a small spoon) and use of <1.5 mL reagent volume which was >70 times less than that applied in the conventional method. Moreover, with the presence of potential interference ions, greater selectivity towards magnesium was observed. Furthermore, the reagents used in our developed test kit were stable for >6 months at room temperature. The results obtained on real samples were in agreement with those obtained from the conventional complexometric titration (ISO 17403: 2014(E)) method. The proposed technique provides a low-cost, rapid, simple, selective and on-site analysis of magnesium content in NRL.

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1. Introduction

Para rubber tree (*Hevea brasiliensis* Muell. Arg.) is a very important economic plant in Thailand where over three million tons a year of Natural Rubber Latex (NRL, the product of Para rubber tree) is exported [1,2]. The major component in NRL is *cis*-1,4-polyisoprene with non-rubber components such as carbohydrates, proteins and lipid based medium for bacteria growing. pH values of normal NRL solution range from 6 to 7 [3,4]. Under this condition, the surface of NRL particles will become negatively charged due to the presence of carboxylate ions of protein (Alpha-globulin with pI of 4.8) and the hydrolysis of R-Lecithin phospholipid on the NRL surface [4] (Fig. 1a). A critical step in production of NRL with high quality is identification of NRL components.

Magnesium (Mg²⁺) is one of the most important components suppressing NRL performance and quality, e.g. by direct interaction of Mg²⁺ with the carboxylate ions contained in the NRL. This produces insoluble, un-hydrated and un-ionized magnesium soap and insoluble magnesium hydroxide in the aqueous phase. Both of these phenomena invariably cause destabilization of NRL. Furthermore, Mg²⁺ ions can form primary valence linkages between the interface of adjacent latex particles. This can initiate flocculation and further lead to destabilization of NRL [5] Fig. 1b.

Mg²⁺ concentration is limited to within 40 mg L⁻¹ prior to distribution to manufacturers such as glove and condom companies [6]. Normally, removal of Mg²⁺ in rubber is performed based on a precipitation reaction, e.g. by addition of excess amount of ammonium phosphate (>5 times of Mg²⁺ concentration) to rubber latex solution. Alternatively, diammonium hydrogen phosphate (DAHP) can be used for precipitation of Mg²⁺ with the related reaction shown in Eq (1) [7,8] and Fig. 1c.

* Corresponding author.

E-mail address: purim.j@ubu.ac.th (P. Jarujamrus).

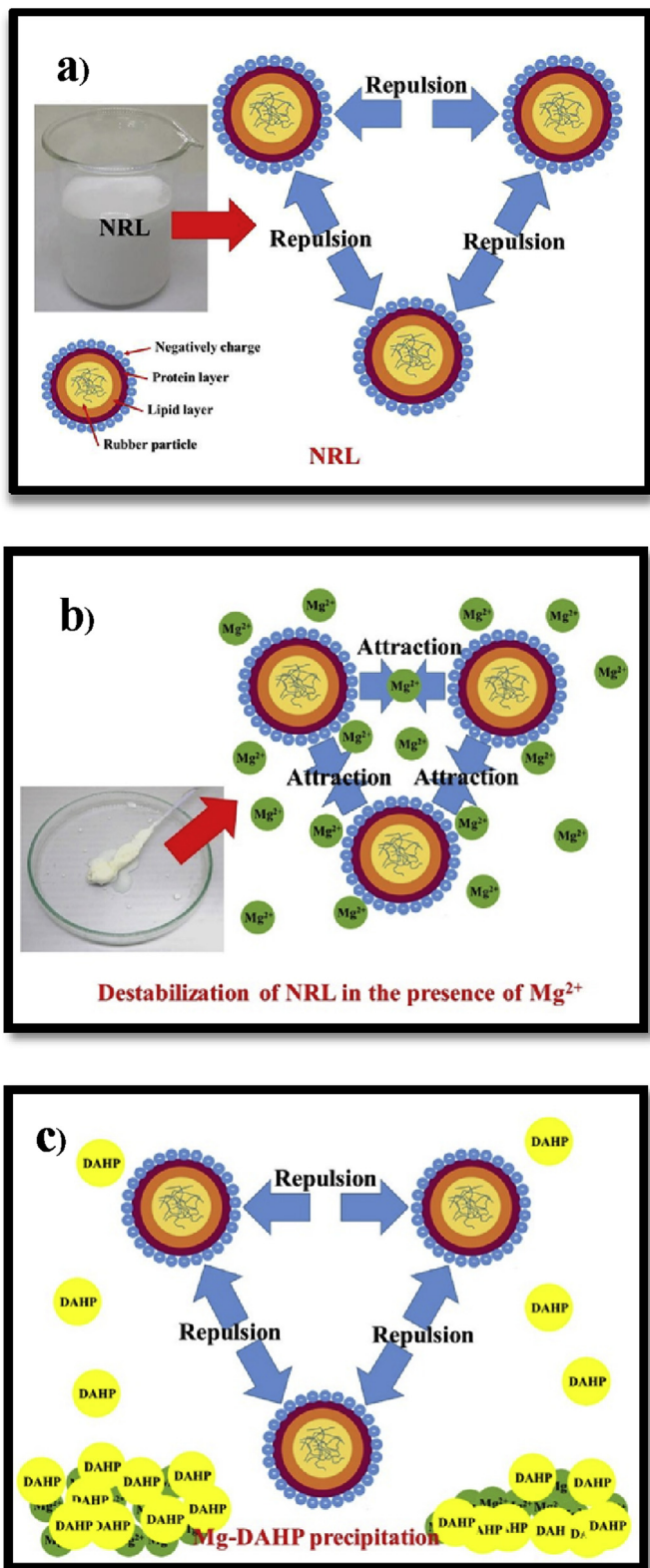
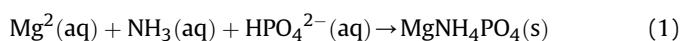
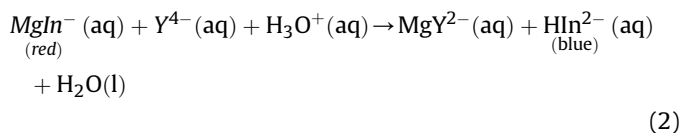


Fig. 1. Carboxylate ions contained in the NRL a) the proposed mechanism between Mg²⁺ ions and NRL b) Elimination of Mg²⁺ by adding DAHP into NRL c).



A conventional approach involves Mg²⁺ analysis in NRL based

on the complexometric titration with ethylenediamine tetraacetic acid (EDTA, H₂Y²⁻), disodium salt (soluble form) using eriochrome black T (EBT) as an indicator. Since dissociation of EDTA disodium salt and EBT depends on pH of the medium, addition of ammonium chloride (NH₄Cl)/ammonium hydroxide (NH₄OH) buffer solution is required in order to control pH of the solution to be ≥ 10, facilitating reaction between EBT and Mg²⁺. At the end point of the reaction, the solution color changes from red to blue, according to the reaction shown in Eq (2) [8], for Mg²⁺ analysis in NRL.



Apart from the well controlled pH of the solution facilitating interaction between Mg²⁺ and EDTA, a masking agent is also added to prevent foreign ions, such as potassium (K⁺), sodium (Na⁺), calcium (Ca²⁺), zinc (Zn²⁺), iron (Fe³⁺), copper (Cu²⁺) and manganese (Mn²⁺) interfering complexation between Mg²⁺ and EDTA in NRL. Potassium cyanide (KCN) is a common masking agent used in the standard method [9,10]. However, it is well known that cyanide compounds are very toxic. Therefore, development of a cyanide-free method for determination Mg²⁺ in NRL is still a challenge.

Satheinperakul et al. (2008) [11] have reported methods for magnesium determination in natural rubber latex based on the potentiometric titration with a Hg-EDTA electrode. Masking agents were not applied since they disturbed the end point of the titration. Their methods showed linearity range for Mg²⁺ detection from 36 to 126 mg L⁻¹, which is in good agreement with the results obtained from atomic absorption spectrometry. Unfortunately, they found that the presence of zinc and cadmium at high levels interfered with the determination of Mg²⁺. In 2011, a method and composition for quantifying magnesium ions based on conventional complexometric titration was patented (WO2011139245) using NaHS as a selectively precipitating interfering metal ions in NRL instead of using KCN as mentioned above [12]. Afterwards, ISO 17403: 2014(E), Rubber-Determination of magnesium content of field and concentrated natural rubber latices by titration (cyanide-free method by using NaHS as a masking agent) [13–15] was established in 2014. However, the reported approaches are still complicated in terms of multiple steps of analysis including need for sample pretreatment before analysis, reagent volume consumption (>105 mL), resulting in more waste and potential interference ions in NRL if a masking agent was not applied (Table 1.). Moreover, many reagents are needed for analysis which also requires skill to perform in the laboratory.

Nowadays, two types of test kit for magnesium detection based on colorimetry have been commercially available. One is the field test kit in seawater based on complexometric titration without addition of masking agents. A limit of detection (LOD) is found within the range of 15–100 mg L⁻¹ [16–19]. Moreover, interference from calcium and strontium was not observed. Another type of the commercial test kit is based on an enzymatic assay performed in a 96 well flat-bottom plate coupled with spectrophotometer detection at 450 nm [20]. The assay involves specific interaction between glycerol kinase enzyme and Mg²⁺ which results in a linear range of 1.5–7.5 mg L⁻¹ without interference from foreign ions such as Fe²⁺, Cu²⁺, Ni²⁺, Zn²⁺, Co²⁺, Ca²⁺ and Mn²⁺. However, the approach as mentioned above is costly and requires expertise to perform (Table A). A miniaturized complexometric titration between Mg²⁺ and EDTA is thus considered to be a key reaction in this development.

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