การตรึงสารสกัดมันเทศสีม่วงในฟิล์มยางธรรมชาติ: ศักยภาพในการประยุกต์ใช้ตรวจวัดไอออนเหล็ก

Immobilization of Purple Sweet Potato Extract in Natural Rubber Film: A Potential Application for the Detection of Fe(III)

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บทคัดย่อ

งานวิจัยนี้เป็นการพัฒนาฟิล์มเซนเซอร์สำหรับการตรวจวัดไอออนเหล็ก Fe(III) โดยการตรึงสารสกัดจากมันเทศสีม่วงในฟิล์มยางธรรมชาติ วิเคราะห์ลักษณะของฟิล์มโดยใช้เทคนิคฟูเรียร์ทรานส์ฟอร์มอินฟราเรดสเปกโตรสโคปี (Fourier Transform Infrared Spectroscopy) และวิเคราะห์ความเข้มสีโดยใช้เครื่องวัดสี จากผลการวิเคราะห์ พบว่าสามารถตรึงสารสกัดจากมันเทศสีม่วงในฟิล์มยางธรรมชาติได้ และเมื่อนำฟิล์มยางธรรมชาติที่ตรึงสารสกัดจากมันเทศสีม่วงไปตรวจวัดไอออนเหล็ก พบว่ามีการเปลี่ยนแปลงสีของฟิล์มจากสีตะไคร่น้ำเป็นสีเหลืองที่สามารถสังเกตได้ด้วยตาเปล่า และการเปลี่ยนแปลงความเข้มสีสอดคล้องกับความเข้มข้นของสารละลายโลหะตั้งแต่ 12.5 ถึง 100 มิลลิกรัมต่อลิตร โดยใช้เวลาในการแช่ 2 ชั่วโมง ดังนั้นจะเห็นได้ว่าฟิล์มยางธรรมชาติที่มีสารสกัดจากมันเทศสีม่วงที่พัฒนาขึ้นมีศักยภาพในการพัฒนาสำหรับการตรวจวัดไอออออนเหล็กได้

คำสำคัญ: เซนเซอร์ สารสกัด มันเทศสีม่วง ไอออนเหล็ก ฟิล์มยางธรรมชาติ

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Abstract

A film sensor for the detection of Fe(III) was developed by the immobilization of the extract from purple sweet potato in films produced by natural rubber latex. The films were characterized by using Fourier-Transform Infrared Spectroscopy (FTIR), and the color intensity was measured using a color intensity meter. The analysis of film found that the purple sweet potato extract can be immobilized in the natural rubber latex film. The films exhibited an irreversible color change from moss to yellow in response to the presence of Fe(III) at two-hour contact time, which was correspondent to the concentration of a metal solution from 12.5 to 100 ppm. Therefore, it can be seen that the developed natural rubber film containing the purple sweet potato extracts has the potential to develope as sensor for the determination of iron (III).

Keywords: Sensor, Extract, Purple sweet potato, Iron, Natural rubber film

Introduction

Iron is the fourth most abundant element in the earth's crust and the most abundant transition metal in the human body (Weber et al., 2006). It supports a range of physiological processes such as oxygen transport, electron transfer, respiration, and gene expression (Dixon & Stockwell, 2014). Iron deficiency leads to anemia (Kew, 2014), whereas excess iron can increase the production of reactive oxygen species, resulting in oxidative stress cascades that lead to lipid oxidation and DNA damage (Dixon & Stockwell, 2014; Chang, 2015). The determination of Fe(III) is traditionally conducted using methods such as inductively coupled plasma optical emission spectrometry (Rahman et al., 2012), flame atomic absorption spectrometry (Mashhadizadeh et al., 2008), and inductively coupled plasma mass spectrometry (Martinez-Lopez, Sakayanagi, & Almirall, 2018). Although these methods provide high sensitivity with relatively low detection limits, disadvantages include operation costs and high equipment acquisition, as well as difficulty in performing in situ analyses. As an alternative, the use of sensors can provide simple and inexpensive detection of the metal (Piriya et al., 2017).

There are various methods to determine iron metal ions, such as photometric and electrometric methods. In addition, most of these methods require expensive equipment and time-consuming, trained operators. The determination of metal ions in environmental water samples by using inexpensive measurements is widely interesting. To meet this challenge, color development is one of the popular methods that have been investigated. Various reagents used as chelating agents for the determination of metal ions must be synthesized, and it is usually not an environmental friendly process. Moreover, the synthesized compounds would be costly.

Anthocyanins are water-soluble pigments most commonly present in flowers, fruits, and vegetables. They are responsible for the red, blue, and purple coloration of plants (Chaiyasut et al., 2016). They have considerable potential for use in the development of colorimetric sensors, due to their ability to change color according to changes in the pH of the medium or by complexation with metals ions (Khaodee et al., 2014; Khaodee et al., 2018; Majdinasab et al., 2018). It is found in many plants such as flower of roses (Ogata et al., 2005), berries (Kang et al., 2021), red cabbage (Khaodee et al., 2014; Majdinasab et al., 2018) including purple sweet potato (Khaodee et al., 2018; Wulandari et al., 2018).

Various polymer matrices have been used as supporting materials for the colorimetric sensor, with a selection of the matrix being dependent on the intended application of the sensor (Kaur et al., 2018; Nam et al., 2018). Natural rubber (NR) is known for its excellent tensile strength and elongation properties and becomes one of the most important materials for tires, gloves, and other NR products. The application of NR may be explored further once it is subjected to modification. Samples of modified NR, such as liquid natural rubber (LNR), degraded and shorter NR chains have been shown to be useful as compatibilizers (Mounir et al., 2004; Dahlan et al., 2002), reactive plasticizers (Srilathakutty et al., 1996), adhesive (Wayakron et al., 2013) and coating (Mathew et al., 2010).

The objective of this study was to create an environmentally friendly colorimetric sensor for detecting Fe(III) by immobilizing purple sweet potato extract in natural rubber latex films.

Materials and Methods

2.1 Preparation of the natural rubber latex films with the purple sweet potato extract

The purple sweet potato extract powder was purchased from Kenko, Japan. 60 % concentrated latex is fresh latex treated with a 60 % concentrated ammonia solution purchased from LB Science. The natural rubber latex films were produced using the casting technique. The films were synthesized from natural rubber (NR) in the form of concentrated latex at 60% by weight since it has high latex as well as low protein, carbohydrate, and nitrogen (Ali Shah et al., 2013). To prepare the films, the concentrated latex 60% (w/w) was mixed with vulcanizing chemical reagents as shown in Table 1. The mixing solution was prepared by mixing concentrated latex 60% (w/w) with sulfur, zinc diethyl-dithiocarbamate (ZDEC), zinc-2-mercaptobenzothiazole (ZMBT), Wingstay L, zinc oxide, and propylene glycol, respectively. Individual chemicals were mixed into the solution after 5 minutes of stirring at 75°C. The purple sweet potato powder (1, 3, and 5 g) was added and stirred for 10 minutes. The 3 g of the resulting solution were poured onto the prepared petri dish containing 9.0 cm in diameter, followed by drying in an oven at 50 °C for 24 h. The films were cut into 1x1 cm^2 pieces and stored in a desiccator until used. The characterization was performed by Fourier Transform Infrared Spectroscopy.

Table 1 The natural rubber latex films formulation.

Ingredients	Mass (g)	Functions
50 % Sulfur	0.8	rubber stabilizer
50 % ZDEC	0.4	rubber maturing accelerator
50 % ZMBT	0.4	rubber maturing accelerator
50 % Wingstay L	0.4	rubber deterioration
50 % Zinc oxide	1.8	catalyst stimulant
33 % DPG	0.4	rubber maturing accelerator

2.2 Evaluation of the purple sweet potato extract solution as sensors for iron

The buffers pH 1-2 were prepared from hydrochloric acid-potassium chloride, pH 3-6 were prepared from acetic acid-sodium acetate, pH 7-9 were prepaed from hydrochloric acid-disodium hydrogen phosphate, pH 10-11 were prepared from sodium hydroxide-disodium hydrogen phosphate and pH 12 was prepared from sodium hydroxide-potassium chloride. All buffers were prepared with deionized water treated with a reverse osmosis deionized system (Millipore, Bedford, USA). To find out the detection pH condition that is selective to iron, the pH of the buffer was first tested. The color-developing method was observed by adding 100 µl of buffer pH 1-12 and 40 µl of Fe(III) solutions at 100 ppm, followed by 20 µl of purple sweet potato extract solution. The color change was observed with the naked eye at different pH related to the iron-purple sweet potato extract formation complexes.

2.3. Evaluation of the natural rubber films with the purple sweet potato extract as sensors for iron

The time of contact of the film with the metal solution was an important parameter that should be considered in establishing the best conditions for use of the sensor. In order to evaluate the films containing the purple sweet potato extract as sensors for Fe(III), they were immersed in 5 mL of 50 ppm Fe(III) solutions at pH 11.0 for 30 min, 1 h, 2 h, and 10 h at 25 °C. After exposure, the films were collected and washed with deionized water. The excess water was removed using filter paper and the color of the central region of the film was measured using a colorimeter (CR-10, Konica Minolta, Japan). The CIE (Commission Internationale de l'Eclairage) color system was used, obtaining the parameters L*, a*, and b*. The experiments were performed in three replications.

Results and discussions

3.1 Incorporation of the purple sweet potato extract into the natural rubber latex film

Low solubility in aqueous solutions is an important property of solid sensors because it can prevent the loss of the sensor molecule during the identification of the analyte. Fiure 1 shows the natural rubber films (a) with and without purple sweet potato extract. The film remained homogeneous in the presence of the natural dye, with an intense moss color indicating that the purple sweet potato extract had been incorporated into the film (b-d). The different concentrations (1, 3, and 5 g) of purple sweet potato extract provided different colored films and were less homogeneous when the concentration of purple sweet potato extract increased.

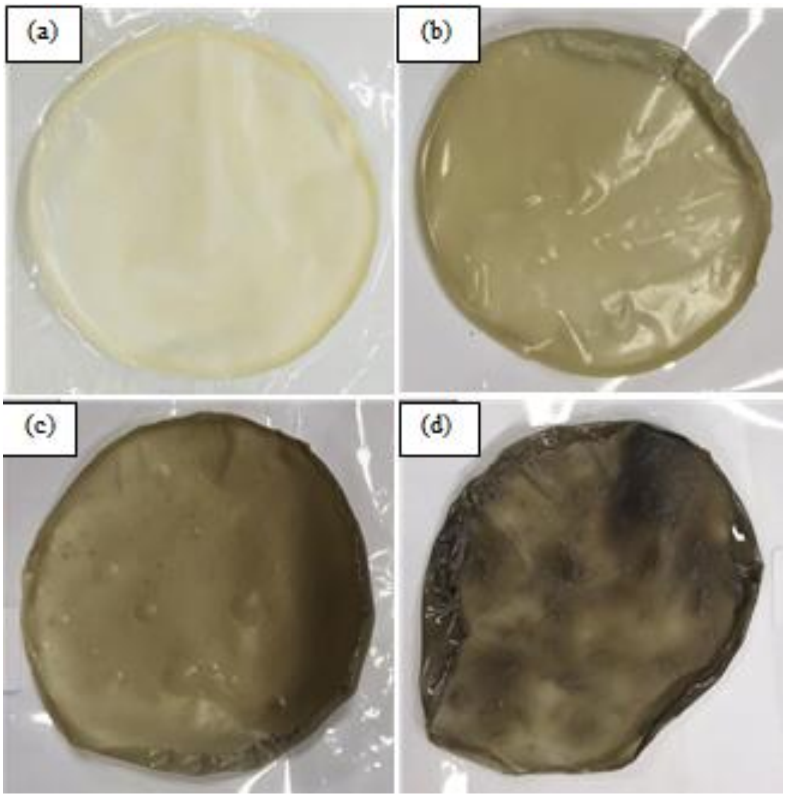


Figure 1. Films produced using a natural rubber, without (a) and with (b) 1 g of purple sweet potato extract, (c) 3 g of purple sweet potato extract and (d) 5 g of purple sweet potato extract.

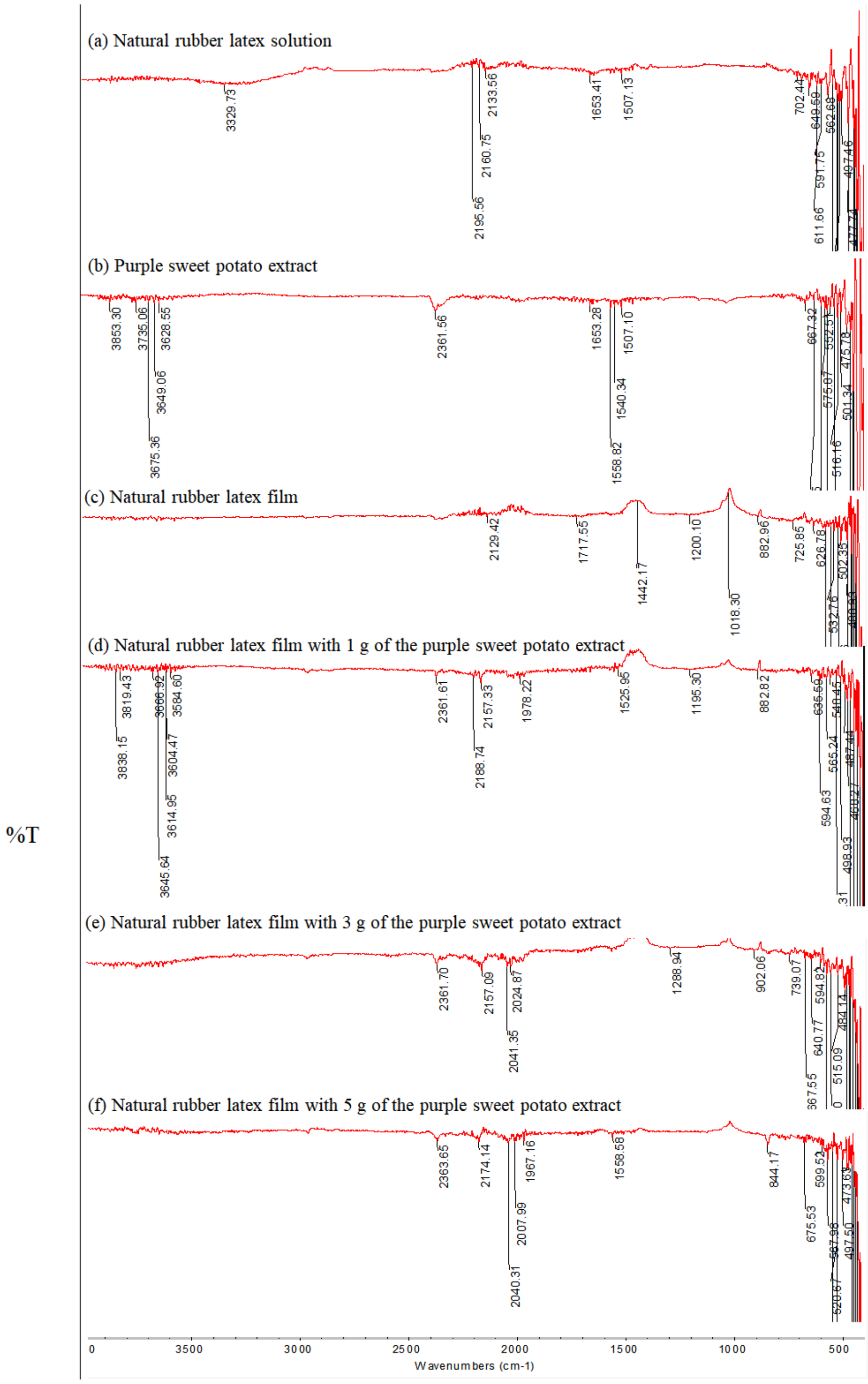


Figure 2. FTIR spectra of (a) the natural rubber latex solution, (b) the purple sweet potato extract, (c) the natural rubber latex film without purple sweet potato extract, (d) the natural rubber latex film with 1 g of the purple sweet potato extract, (e) the natural rubber latex

film with 3 g of the purple sweet potato extract, and (f) the natural rubber latex film with 5 g of the purple sweet potato extract.

In the spectra for the purely natural rubber latex solution (Figure 2a), a broad band centered at 3329.73 cm^{-1} was observed, which can be assigned to the stretching mode of the hydroxyl groups of the natural rubber latex chains. A band at 1653.41 cm^{-1} was due to C=C stretching vibration. Characteristic bands of natural latex at around 702.44 cm^{-1} could be attributed to C-H stretching vibration. The results are consistent with other research (Ibrahim et al., 2014). The spectra of the purple sweet potato extract (Figure 2b), a peak at 3628.55 cm^{-1}, 1653.28 cm^{-1}, and 1507.10 cm^{-1} which were caused by the elongation of the -OH, C=C, and C-C bonds of the phenol group of cyanidin anthocyanins. These peaks showed the characteristics of the aromatic rings of flavonoid compounds. The results are consistent with other research papers (Silva-Pereira et al., 2015). The same bands, with small shifts, were observed in the spectrum of the natural rubber latex film with the purple sweet potato extract at different concentrations of pigments, with small peaks at 2500–2300 cm^{-1} due to the vibrations of the carboxylic group. It can be seen that the bands of the purple sweet contain films (Figure.2d-f).

3.2 Evaluation of the purple sweet potato extract solution as sensors for iron

The pH of the solution is a major parameter affecting the purple sweet potato extract. The pigment solution appeared in a pink-red color at pH 2–4, purple at pH 5–7, blue at pH 8–10, and green at pH 11–12. In buffer solutions, the complexation of purple sweet potato extract and Fe(III) was investigated. The purple sweet potato extract clearly formed complexes and the color change can be observed by the naked eye at pH 4–12 as shown in Figure 3. After the chelating agent interacted with the metal ion, the bathochromic shift occurred due to the interaction between the metal ion and the ortho-dihydroxyl group at the B-ring of the cyanidin molecule (Khaodee et al., 2014), as shown in Figure 2(b). Therefore, pH 11.0 was chosen for further analysis of iron (III) using the film sensors.

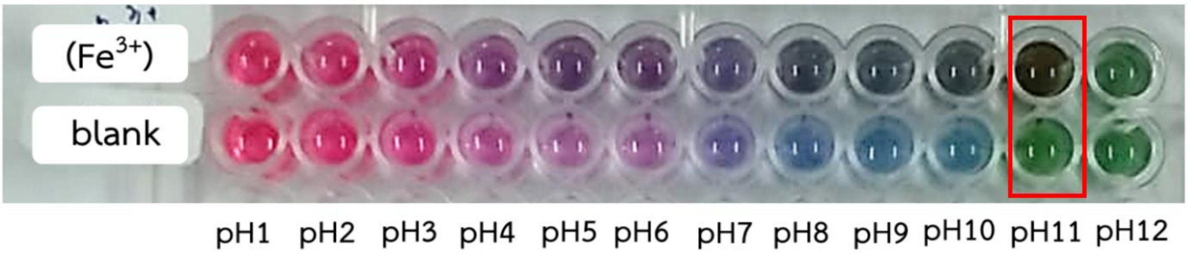


Figure 3. Qualitative determination of Fe(III) at different pH buffer solutions.

3.3 Evaluation of the natural rubber films with the purple sweet potato extract as sensors for iron

Purple sweet potato extracts are widely used as pH sensors because they can change color according to the concentration of hydrogen ions in the metal medium (Wulandari et al., 2018; Choi et al., 2017; Ma and Wang, 2016). Moreover, the time of contact of the film with the metal solution was an important parameter that should be considered in establishing the best condition for use of the sensor. The contact time of the film sensor to the ferrous metal solution was studied, as shown in Table 2.

Table 2 Images of the films after exposure to the blank and Fe(III) solutions with difffferent contact times at 25 °C.

Film types	Solution tested							
	Blank				50 ppm Fe(III)			
	30 min	1 h	2h	10h	30 min	1 h	2h	10h
NR film								
1g PSPE-NR composite film								
3g PSPE-NR composite film								
5g PSPE-NR composite film								

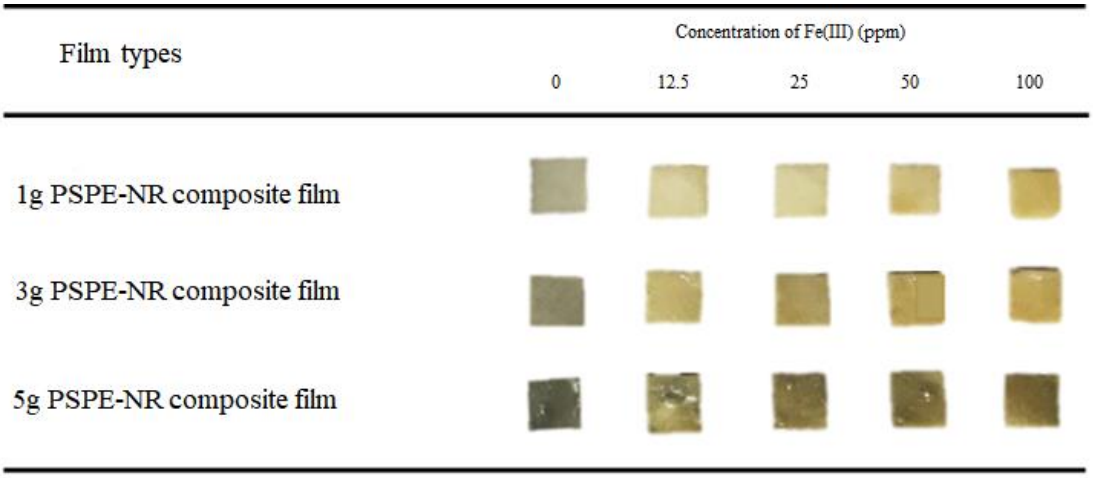
There was no color change of the natural rubber latex film under the blank and the 50 ppm Fe(III) solution when the contact time increased, as well as the natural rubber latex film with the purple sweet potato extract at different concentrations in the blank solution when the contact time increased from 30 min to 10 h. There is a significant difference when the natural rubber latex film with the purple sweet potato extract is immersed in a solution of 50 ppm Fe(III). The change in color indicates the interaction between the anthocyanin in the pigment of the purple sweet potato extract and the ferrous metal solution. This might be because anthocyanin has a dihydroxy group. Therefore, it is possible to create complex assemblies or react between metals. (Castaneda-Ovando et al., 2009; Li et al., 2016). Therefore, a contact time of 2 h was chosen to continue the study under the conditions of a buffer solution of pH 11. It was clear that the contact time of the film in the metal solution was an important parameter.

In order to evaluate the capability of the developed sensor to identify iron (III), colorimetric analyses of the film were performed after its immersion in metal solutions. The color of the film was determined by the nature of the cation, while the intensity of the color depended on the time of contact of the film with the metal solution. The intensity of the natural rubber latex film in the blank and 50 ppm Fe(III) was the same over the times of contact. The films maintained a white color, with a high intensity of the parameter L*. On the other hand, the natural rubber latex containing the purple sweet potato extract film maintained a moss coloration in the blank solution, with a higher intensity of the parameter L*, compared to the film immersed in Fe(III) solution. This was reflected in significant changes in parameter a*, together with small changes in parameter b*. The results are presented in Table 3.

Table 3 Colorimetric parameters obtained for the films after immersion in blank and 50 ppm Fe(III) solutions at the contact time of 2 h.

Film types	Solution tested					
	Blank (contact time, 2h)			50 ppm Fe(III) (contact time, 2h)		
	L*	a*	b*	L*	a*	b*
NR	87.9 ± 0.66	1.42 ± 0.12	11.41± 0.70	87.50± 1.48	1.77 ± 0.25	11.98 ± 5.65
1 g PSPE-NR	81.46 ± 0.89	0.42 ± 0.11	12.06 ± 1.49	71.04 ± 1.06	1.50 ± 0.38	18.73 ± 3.04
3 g PSPE-NR	66.20 ± 4.12	1.63 ± 0.49	17.20 ± 4.40	56.77 ± 0.11	1.51 ± 0.06	13.13 ± 0.12
5 g PSPE-NR	58.06 ± 5.97	1.33 ± 0.56	11.64± 3.48	42.60 ± 3.17	1.75 ± 0.63	10.86 ± 1.38

Table 4. Images of the films after immersion in metal solutions containing Fe(III) at difffferent concentrations.



Film types	Concentration of Fe(III) (ppm)				
	0	12.5	25	50	100
1g PSPE-NR composite film					
3g PSPE-NR composite film					
5g PSPE-NR composite film					

The purple sweet potato extract composite with the natural rubber latex films exhibited an irreversible color change from moss to yellow in response to the presence of Fe(III) by the naked eyes, corresponding to the concentrations, from which it can be concluded that the prepared sensor films can analyze ferrous metal solutions. The images of the color changes were observed by the naked eye and are presented in Table 4.

Suggestions

1. The application of purple sweet potato extract sensor film with other metals.
2. Study the effect of interferences and real sample analysis.
3. Study the film morphology using scanning electron microscope (SEM) and film thickness.

Conclusions

This research was to study the synthesis of the films from natural rubber latex composited with purple sweet potato extract. The functional group analysis by FTIR technique was able to confirm the preparation of the purple sweet potato extract contained in the rubber film, and when applied as a colorimetric sensor for ferrous metal solution analysis, it was found that the color change of the films from moss to yellow was related to the concentration of the metal solution, indicating that the developed sensor film was able to detect the amount of ferrous metal. This developed technique is easy to use, convenient, and fast. The materials used were derived from natural extracts and are non-toxic and environmentally friendly.

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