10th Eco-Energy and Materials Science and Engineering Symposium


On December 5-8, 2012
Sunee grand hotel,
Ubon-ratchathani

Organized by

Co-organized by
PREFACE:
Message from the President of
Rajamangala University of Technology Thanyaburi

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This international conference is not only giving an opportunity for Thai and foreign researchers to present and discuss their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Our program consists of six research tasks: (1) Energy Technology, (2) Environmental and Social Impact, (3) Nanotechnology and Materials Science, (4) Energy Economics and Management, (5) New Energy technology and (6) Nuclear Technology.

I would like to take this opportunity to express our sincere gratitude to our two distinguished Plenary Speakers for kindly accepting our invitation. I deeply appreciate of the very strong support given by Kyoto University. Thanks to the tireless works of the Organizing Committee, the Technical Program Committee, the invited speakers and paper and poster contributors, and excellent program been assembled to cover a broad spectrum of interesting topic.

We warmly welcome you to the 10th EMSES on December 5-8, 2012, Ubon Ratchathani, Thailand.

Numyoot SONGTHANAPITAK, Ph.D.
President of Rajamangala University of Technology Thanyaburi
and Conference Chairman of 10th EMSES 2012
PREFACE:
Message from the Director of
Institute of Advanced Energy, Kyoto University

It is my great pleasure to have the 10th Eco-Energy and Materials Science and Engineering Symposium (EMSES) with Rajamangala University of Technology Thanyaburi (RMUTT) under the long-term collaboration between RMUTT and Kyoto University. The 1st EMSES was held in 2001 in Thailand and the symposium has been expanded in its scientific contents as well as the academic network. I believe that the 10th EMSES gives a good opportunity to all participants to exchange their knowledge and idea to realize eco-friendly energy system in society. I would like to express my welcome to all participants and sincere thanks to the 10th EMSES organizing committee and all supporting organizations to make us having this symposium.

I hope that the symposium will be successful and lead to further progress in energy science and technology and also in friendships of participants.

Professor Yukio Ogata, Ph.D.
Director of Institute of Advanced Energy, Kyoto University
PREFACE:
Message from the Former Dean of
Graduate School of Energy Science, Kyoto University
Program Leader,
Global COE “Energy Science in the Age of Global Warming”

I want to express my hearty welcome to all participants of Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This symposium is aiming the realization of importance of energy and materials technology through the academic, science and technology network among the world communities. The symposium gives an opportunity for researchers to discuss their research works and also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the further cooperation work will be developed.

I would like also extend my sincere thanks to all who made the meeting possible, including the 10th EMSES organizers, the SEE forum committee members, and the Japanese Government, JSPS, for their kind support. I am looking forward to seeing you in Ubon Ratchathani, Thailand.

Professor Takeshi YAO, Ph.D.
Former Dean of Graduate School of Energy Science, Kyoto University
and Program Leader, Global COE “Energy Science in the Age of Global Warming”
Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES).

RMUTT has a major mission on encouraging and supporting all areas of research. One of the key reasons is to assist in developing capability in science and technology in order to cope with recent rapid change in this field. We have jointly set up an academic symposium on the 10th EMSES with the perception on the significance of exchanging knowledge and research experiences between researcher in the field of energy, materials technology and environmental science. This symposium is not only giving an opportunity for Thai and foreign researcher to present and discussion their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the closer future cooperation incorporate with joint-research works will be developed. Thus, to support the aforesaid role, the symposium working committee would like to invite you to participate in this academic symposium.

I would like to express our sincere thanks to the organizing committee, participants and contributors for your kind corporation to this symposium. I wish this symposium proceeding will be a useful reference for future scientific research development.

Sommai PIVSA-ART, Ph.D.
Dean of Faculty of Engineering, RMUTT
Director of CoE on Sustainable Energy System (Thai-Japan)
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**Area: Energy Technology (ET):**

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**Area: New Energy Technology (NT):**

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Katsue N. Ishihara

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Preparation and Characterization of Natural Rubber/Poly [styrene-co-2-(methacryloyloxy) ethyl trimethylammonium chloride] Nanocomposites by Heterocoagulation

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Abstract – The simple technique as heterocoagulation was used to prepare natural rubber (NR) with poly[styrene-co-2-(methacryloyloxy) ethyl trimethylammonium chloride] (P(S-QDM)) nanocomposites (NR/P(S-QDM)). P(S-QDM) particle was prepared by emulsion polymerization at 80°C using azobisisobutyramid dihydrochloride as initiator. Under the alkaline condition, NR latex (NRL) surface represents negative charge deriving from protein adsorbed on its surface while strong positive charge deriving from QDM structure is obtained. The assembly via electrostatic interaction between NRL and P(S-QDM) particles in the emulsion state is then taken place with pH adjustable from 2 to 8. The particle surface, particle size and charge of the polymer nanocomposites were measured by scanning electron microscope, dynamic light scattering and zeta potential, respectively, to compare those with the original properties.

Keywords: Natural Rubber, Nanoparticle, Nanocomposite, Electrostatic interaction, Heterocoagulation

1. INTRODUCTION

Natural rubber latex (NRL) is one of the most important economic products of Thailand. Because it is able to comfortably improve the properties with the addition of appropriate amount of fillers to benefit the application concerned [1], it is widely used in various applications such as medical glove and tubing [2-4]. Polymer nanocomposites as only small amount of nanometer size filler dispersed inside the polymer exhibit markedly improved properties compared to the original or their traditional composites. They represent an alternative and a powerful technique to improve various polymer properties including increased modulus and strength, outstanding barrier properties, improved solvent and heat resistance and decreased flammability [5]. To improve natural rubber (NR) properties, various techniques have been done as adding inorganic and organic materials such as carbon black [6] ultra-fine calcium carbonate [7] and modified montmorillonite [8]. The heterocoagulation is a simple and interesting technique to prepare composite materials. The materials as the small and large particles in the emulsion state are blended by various forces such as electrostatic and hydrophobic interactions. It can be used to prepare various composite materials such as inorganic-inorganic [9], inorganic-organic [10] and organic-organic [11-15] composites.

In our previous work [16], NR/poly(styrene-methacrylic acid) (P(S-MAA)) and NR/poly(styrene (PS) nanocomposites were successfully prepared by heterocoagulation using electrostatic interaction. NRL and vinyl polymer (PS and P(S-MAA)) particles were blended in the emulsion state at pH of 1 where the opposite charges between those polymers were obtained. P(S-MAA) and PS represent negative charge (mainly derived from potassium persulfate initiator) on their surfaces while NRL surface showed the positive charge. The mechanical properties of the obtained polymer nanocomposites are dramatically improved. However, less positive charge of NRL (+17.1 mV) leading to less dynamic to interact with the negative charge of the polymer nanoparticles resulting in the formation of unstable polymer nanocomposites. To overcome this problem, pH of the blending condition was changed from acidic (pH of 1) to alkaline (pH of 8) condition to introduce highly negative charge on NRL surface. In addition, poly[styrene-co-2-(methacryloyloxy) ethyl trimethylammonium chloride] (P(S-QDM)) particle having strong positive charge was used instead of PS and P(S-MAA) particles[17]. The obtained NRL/P(S-QDM) nanocomposite represents good colloidal property with long term storage. In this work, the extension study including NRL and P(S-QDM) ratios and pH of pre-blending in the heterocoagulation step were optimized.

2. EXPERIMENT

Materials

High ammonia NR latex (ca. 60% dry rubber content; donated from Thai Rubber Latex Co., Ltd., Bangkok, Thailand) was used as received. Styrene (S; Aldrich; purity, 99%) was purified by pass through the column packed with basic aluminium oxide. 2-(methacryloyloxy) ethyl trimethylammonium chloride (QDM; Aldrich) was used as received. Azobisisobutyramid dihydrochloride (AIBA; Wako) was used as received. The chemical structures of QDM and AIBA are shown in Fig. 1. Analytical grade of sodium hydroxide (NaOH; BDH Prolabo), hydrochloric acid (HCl; Ajax Fineschem) and polyoxyethylene (20) sorbitan monooleate (Tween 80; Aldrich) and cetyltrimethyl ammonium bromide (CTAB; Fluka) were used as received.
Fig. 1. The chemical structures of 2(Methacryloxyloxy) ethyltrimethyl ammonium chloride (a) and AIBA (b)

**Preparation of polymer nanoparticles**

(P(S-QDM)) nanoparticles were prepared by emulsion polymerization under the conditions listed in Table 1. The water (129 g) containing surfactant (1 g of Tween80) was charged into the reactor. After the addition of monomers (15 g of S and QDM), the reaction was purged with \( \text{N}_2 \) for 30 min. Consequently, the polymerization was initiated by the addition (5 g) of AIBA aqueous solution (1.2 wt % of monomer) and carried out at 80°C with a stirring rate of 200 rpm for 8 hours.

Table 1. Recipe for the preparation of styrene (S)- 2- (methyleneoxyloxy) ethyltrimethyl ammonium chloride [P(S-QDM)] nanoparticles by emulsion polymerization

<table>
<thead>
<tr>
<th>Ingredients</th>
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<tbody>
<tr>
<td>Styrene (g)</td>
<td>13.50</td>
</tr>
<tr>
<td>QDM (g)</td>
<td>1.50</td>
</tr>
<tr>
<td>AIBA (mg)</td>
<td>60.00</td>
</tr>
<tr>
<td>Tween80 (g)</td>
<td>1.00</td>
</tr>
<tr>
<td>( \text{H}_2\text{O} ) (g)</td>
<td>134.00</td>
</tr>
</tbody>
</table>

200 rpm, 80°C, 8 hr., purged by \( \text{N}_2 \)

**Preparation of NRL/P(S-QDM) nanocomposites**

The nanocomposites of NR/P(S-QDM) were prepared using electrostatically driven heterocoagulation technique (Fig. 2) according to the following procedures. The pH of NRL aqueous dispersion (solid content about 10 %wt) containing Tween 80 emulsifier (6 %wt of NR) was firstly adjusted from approximately 10 to 2 with 0.3 M HCl. The P(S-QDM) nanoparticle aqueous dispersion (10 %wt solid content at pH of 2) was gradually dropped into the NR solution with mechanical stirring rate of 200 rpm. The polymer composites of NR/P(S-QDM) were obtained when the pH of the mixture solutions were gradually adjusted to 8 by the addition of 0.3 M NaOH. The blending ratio was determined relating to the theoretical number (\( N_{\text{max}} \)) which is the maximum number of small particle [P(S-QDM)] to form a closely packed particulate in a monolayer on a large particle (NRL) as given by Equation (1) shown below [18].

\[
N_{\text{max}} = \frac{2\pi}{3} \left[ \frac{R_L + R_S}{R_S} \right]^2
\]

\( R_L \) radius of large particle

\( R_S \) radius of small particle

**Characterizations**

pH were measured by pH meter multi parameter analyser (pH meter; Consort C831, Belgium) Particle diameters and zeta potential were measured by dynamic light scattering (DLS; Delsa nanoc, Beckman, USA) at 25 °C. Polymer emulsion samples (approximately 10 %wt) withdrawn from the reactor were directly measured by dilution mode of DLS. Scanning electron microscope (SEM; JSM-6340, Jeol Ltd., Japan) were used to investigate the morphology of the P(S-QDM) and NR/P(S-QDM) nanocomposite. For SEM observation, one drop of the polymer dispersion was placed on a nickel SEM stub and dried before Au-coating.

Fig. 2. Schematic of nanocomposite preparation using electrostatically driven heterocoagulation
3. RESULTS AND DISCUSSION

P(S-QDM) nanoparticles were selected to be the small particles having positive charge on their surfaces at alkaline condition (pH of 8). Generally, the obtained positive charge was derived from initiator as AIBA (weak positive charge) and QDM monomer (strong positive charge). However, at the alkaline condition amino group in polymer chain derived from AIBA would be deprotonated. Therefore, the main positive charge represented at the blending condition caused only from QDM monomer. It is according to the above assumption that the prepared P(S-QDM) gives positive charge (> +30 mV) throughout the pH range (1-14) as shown in Fig. 3b. A few additional charges at acidic condition would derive from the protonation of amino group of AIBA. P(S-QDM) nanoparticles (76 nm as shown in Table 2) with narrow particle size distribution (Fig. 4) prepared by emulsion polymerization were the spherical particles as SEM micrograph shown in Fig. 5.

The particles are usually maintain colloidal stability via their surface charges having either lower of -30 or higher of +30 mV [17]. To successfully prepare NR/P(S-QDM) nanocomposites, NRL (particle size of 142 nm as shown in the Table 2) surface should present negative charge in the alkaline condition. Therefore, NRL surface charge was measured at various pHs in order to obtain the information of charge behavior with pH. Before charge measurement, nonionic emulsifier (Tween 80) was added to NRL emulsion to maintain the colloidal stability of NRL throughout the experiment [16, 17]. The charges on its surface varied with the pH from positive to negative charges at acidic to alkaline conditions, respectively as shown in Fig. 3a. Generally, the surface of the NRL was covered with protein molecules. They contain both carboxyl and amino groups showing different charges depending on the pH. At the pH of 8, the negative charge seems to be stable and reached to the maximum (approximately -57 mV). On the other hand, at the same pH, strong positive charge (approximately +40 mV) still obtained on the P(S-QDM) surface. Therefore, pH 8 is selected for the blending condition of both polymer particles. At this condition, they are able to avoid the self coagulation and have sufficient dynamic for the opposite charges interaction. However, in the step of pre-blending, both polymer particles should represent the same (positive) charge to avoid large coalescence of both polymers as mentioned in the previous works [14-16]. Therefore, pH of the system is firstly adjusted to the acidic condition in order to gain positive charge on NRL surface. It is well known that NRL is easily to coagulate and precipitate in the acidic condition. Although, NRL was pre-adsorbed by nonionic emulsifier as Tween 80 to protect the coalescence, some of larger particles or precipitation of NRL with long term storage were still found. The main reason is that the amount of Tween 80 is limited in order to avoid the obstruction of electrostatic interaction of both NRL and P(S-QDM). To successfully prepare NR/P(S-QDM) nanocomposites pH of pre-blending is an important parameter. Therefore, it is further optimized.

![Fig. 3. Zeta potential of NRL adsorbed with nonionic emulsifier (a) and P(S-QDM) (b) at various pH](image)

Table 2. Particle size distributions and Zeta potential of P(S-QDM) nanoparticle prepared by emulsion polymerization and NRL

<table>
<thead>
<tr>
<th>Particle size (nm)</th>
<th>Zeta potential (mV)</th>
</tr>
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<tr>
<td>P(S-QDM)</td>
<td>75.80</td>
</tr>
<tr>
<td>NR-Tween80</td>
<td>142.30</td>
</tr>
</tbody>
</table>

![Fig. 4. Particle size distributions of (——) P(S-QDM) nanoparticle prepared by emulsion polymerization (——) NR-Tween80 and (——) NR/P(S-QDM) prepared by heterocoagulation](image)
Pre-blending condition was studied at various pHs (1-4). The size of NR/P(S-QDM) nanocomposites was increased with the pH as shown in Fig. 6. In the case of pH of 3 and 4, much larger particles (299 and 2,766 nm, for pH of 3 and 4, respectively) were obtained. This seems to the occurrence of NRL self coagulation during the blending competition to electrostatic interaction between NRL and P(S-QDM). In contrast, the size of polymer nanocomposites (175 and 229 nm, for pH of 1 and 2, respectively) is reasonable according to the original polymer particles. Because of less acid, pH of 2 is selected to use for the further study.

Fig. 5. SEM micrograph of P(S-QDM) nanoparticle prepared by emulsion polymerization

Fig. 6. Particle size distributions of polymer nanocomposites at various pHs

To obtain the appropriate amount of P(S-QDM) to form a close-packed monolayer covering the NRL, the ratios of both polymer particles base on N_max were studied. Table 3 represent the particle size and zeta potential of NR/P(S-QDM) nanocomposites at the different ratios of both polymers. The size of the nanocomposites increased with amount of P(S-QDM) from N_max/2 (163.9 nm) to N_max (228.7 nm) and then decreased at the twice of N_max (93.9 nm). This result may accord to the theory that at the N_max the particle size is the largest where P(S-QDM) completely covered NRL by monolayer. In contrast, some of free P(S-QDM) particles were remained in the case of twice of N_max, resulting in the reduction of the average particle size of the nanocomposites. Moreover, the zeta potentials of the nanocomposites at various blending ratios (N_max/2, N_max and, 2N_max) were also measured. It increased with the amount of P(S-QDM) polymer. In the case of the lowest amount of P(S-QDM), the lowest net positive charge (8.74 mV) was obtained because the NRL surface was incompletely covered by P(S-QDM) particles resulting in low colloidal stability. The zeta potential of 23.60 mV was obtained at N_max giving more colloidal stability. Because free of P(S-QDM) particles were presented, the much more positive charge (33.74 mV) was represented. Moreover, NR/P(S-QDM) nanocomposites as nano-cluster in all ratios were found in the SEM micrographs (Fig. 7). The smallest amount of nano-cluster was found in the case of N_max/2 (Fig. 7a) due to insufficient amount of P(S-QDM) particle to envelope the NRL while the others were more observed. The closed-pack monolayer seemed to be formed for N_max without any free P(S-QDM) particles (Fig. 7b). Some of free P(S-QDM) particles were observed in the case of the twice of N_max (Fig. 7c) according to the particle size and zeta potential data. These results indicated that NR/P(S-QDM) nanocomposites were successfully prepared by heterocoagulation technique.

Table 3. Particle size distributions of polymer composites at various blending ratios

<table>
<thead>
<tr>
<th>Particle size (nm)</th>
<th>zeta potential (mV)</th>
</tr>
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<tbody>
<tr>
<td>N_max/2</td>
<td>163.9</td>
</tr>
<tr>
<td>N_max</td>
<td>228.7</td>
</tr>
<tr>
<td>2N_max</td>
<td>93.9</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The NRL/P(S-QDM) was successfully prepared with a simply blending as heterocoagulation via electrostatics interaction. pH of 2 was selected as the pre-blending condition where both NRL and P(S-QDM) represented positive charge. The P(S-QDM) particles were gradually adsorbed on the NRL when pH changed from 2 to 8 resulting in the formation of nanocomposite of NR/P(S-QDM). The nanocomposite seemed to be more stable in the case of N_max than N_max/2 relative to much more positive charge. The additional positive charge of the twice of N_max would derive from free P(S-QDM) particles. Nano-clusters were observed in all cases of NRL and P(S-QDM) ratio. The smallest amount of nano-cluster was observed in the case of N_max/2 while it seemed to be similar in the others. However, more amount of free P(S-QDM) particles was found in the twice of N_max than in N_max.
ACKNOWLEDGEMENTS

This work was supported by The National Research Council, Thailand and partially supported by Thailand Institute of Scientific and Technological Research (given to Ms.Supaporn Promdsorn).

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