



PROCEEDINGS
of
INTERNATIONAL SEMINAR
ON CHEMICAL ENGINEERING
IN CONJUNCTION WITH
SEMINAR TEKNIK KIMIA
SOEHADI REKSOWARDOJO (STKSR) 2017

Clean-Sustainable
Process and Product Technology

2-3rd October 2017
East Hall and West Hall
Institut Teknologi Bandung

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PROCEEDING

**International Seminar on Chemical Engineering
in conjunction with
Seminar Teknik Kimia Soehadi Reksowardojo
(STKSR) 2017**

*“Clean-Sustainable Process and Product
Technology”*

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MESSAGE



Welcome to all honorable keynote speakers, presenters, and participants in the International Seminar on Chemical Engineering in conjunction with Seminar Teknik Kimia - Soehadi Reksowardojo (STKSR) 2017. This annual seminar has been an event of research dissemination and knowledge sharing for chemical engineering community in Indonesian, as well as neighboring countries.

Chemical engineering concern to produce, transform, transport, and properly use of chemical, material, and energy. This field of engineering is therefore closely related to industry development in a country. Process design and analysis of technology, chemical reaction engineering, and safety analysis are the main object of chemical engineering. Chemical engineering has also significant role to fulfill one of millennium development goals, i.e. to ensure environmental sustainability. With limit on resources and increasing environment effect due to industries, we need to move toward a clean-sustainable processing technology.

We encourage youth and motivated professional to develop and share their innovation in the clean-sustainable product and process technology through this seminar. Hopefully, the seminar will bring a productive and fruitful discussion. Lastly, we greatly thanks all sponsors and contributors to support this seminar.

Prof.Dr.Ir. Kadarsah Suryadi, DEA.

Rector of Institut Teknologi Bandung (ITB)

MESSAGE



Dear Colleagues,

On behalf of the Organizing Committee of the International Seminar on Chemical Engineering, I am gladly welcome you all to Institut Teknologi Bandung, Bandung, Indonesia. This year, the Seminar which is held in conjunction with Seminar Teknik Kimia - Soehadi Reksowardojo (STKSR) 2017 raises the topic of 'Clean-sustainable Process and Product Technology'. Through this topic, we are promoting an awareness on global warming issues and sustainable production.

Those topics will be addressed in several categories, i.e. bionergy, chemurgy, alternative energy, fossil energy and mineral processing, process technology, and advance science in plenary lectures and parallel sessions. Here, we encourage young and inspiring keynote speakers to share their energy and experiences. In end of seminar, we invite speakers in a workshop session who will share their industrial/field experiences.

We have prepared social (get-together) event, so that delegates may meet and communicate one another. Finally, the committee gratefully addressed all sponsors and ChemEng-ITB Alumni for funding and supports. We also thank all International/Technical Committee members, all the plenary and invited speakers and all oral/poster presenters for their kind efforts and contributions in making this conference a success.

Thank you

Hary Devianto, Ph.D.

Chairman of STKSR 2017

ACKNOWLEDGEMENT

The completion of this undertaking could not have been possible without the participation and assistance of so many people whose names may not all be enumerated. The contributions are sincerely appreciated and gratefully acknowledged. However, we would like to express our especially deep appreciation and gratitude to the following.

1. Institut Teknologi Bandung, Faculty of Industrial Technology, Department of Chemical Engineering.
2. Lembaga Penelitian dan Pengabdian kepada Masyarakat Institut Teknologi Bandung (LPPM ITB)
3. PT. Petrokimia Gresik
4. Medco Foundation
5. Ir. Rauf Purnama, IPU
6. Biomass Gasification Research Group Department of Chemical Engineering Institut Teknologi Bandung

for their support toward STKSR 2017. We sincerely hope that our good cooperation can extend to other opportunities in the future.

International Seminar on Chemical Engineering
In conjunction with Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) 2017
October 2nd-3rd, Bandung Indonesia



PT PETROKIMIA GRESIK

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Synthesis of Methyl Tin Oxide as Raw Material for PVC Stabilizer

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Abstract

This research is aimed to study the synthesis of methyl tin oxide as a raw material in producing thermal stabilizer for poly vinyl chloride from methyl tin chloride. Ammonium hydroxide and sodium hydroxide were used as bases. The synthesis was carried out in a semi batch stirred vessel. The experimental variables included base concentration (4~9 M), base flowrate (2~8 mL/min), and base excess (0%-20%). It was found that base flowrate did not influence the yield of methyl tin oxide. Stronger base resulted in lower yield of methyl tin oxide. The higher the base concentration, the larger the yield obtained. The optimum base excess was in the range of 5~10%. The tin content of methyl tin oxide obtained was in the range of 62% to 66%, a little bit lower than the maximum theoretical yield. The methyl tin oxide obtained was used to synthesize organotin thermal stabilizer. The thermal stabilizer has been tested using a two rolls mill and was found to be effective in stabilizing poly vinyl chloride.

1. Introduction

Poly vinyl chloride (PVC) is the third largest polymer in volume production in the world¹. The annual production of PVC in Indonesia has reached 20,000 tons. PVC has wide applications and mixes well with additives due to the presence of polar chlorine groups. However, it can be thermally degraded due to autocatalytic dehydrochlorination reaction that start occurring at 70°C. Thermal degradation of PVC is caused by the irregular structures in the form of allylic chloride². It liberates hydrochloric acid so that the chlorines are detached from their bond and leave polyene structures (carbonyl group). This process destroys the backbone of PVC. Thermal degradation lowers the mechanical strength of PVC, PVC changes color when heated, from yellow, brown, and black. Thermal degradation of PVC needs to be prevented as most PVC processing involved heat.

PVC thermal degradation can be retarded by using thermal stabilizers. The thermal stabilizer works as a reactive nucleophile, reacts with the allylic chlorines and scavenge the liberated hydrochloric acid so that the propagation of dehydrochlorination reaction can be terminated³. There are various types of thermal stabilizers, such as lead, barium, zinc, and organotin⁴. Among all types of thermal stabilizers, organotin stabilizer is very potential to be developed in Indonesia. The main raw materials for organotin stabilizers are tin and fat. Both materials are abundant in Indonesia. Indonesia is the second largest tin producer in the world. According to the US Geological Survey, tin reserve in Indonesia reaches 60,000 tons/year. On the other hand, Indonesia as a tropical country is also rich in vegetable oils.

One of the synthesis routes of organotin stabilizers can be described in Fig. 16. The synthesis of organotin stabilizer involves two stages of reactions, namely (1) the synthesis of raw material, and (2) the synthesis of organotin stabilizer. The raw material in the form of methyl tin oxide (MTO) is synthesized from methyl tin chloride (MTC) and a base solution, while the other raw material in the form of mercapto ethyl ester of fatty acid (MEFA) is synthesized through a reaction between fatty acids and mercaptoethanol. At the second stage,

methyl tin oxide and MEFA are reacted to obtain organotin thermal stabilizer. Stage 1 and stage 2 might be carried in separate plants/places. This so called MTO route allows the production of organotin stabilizers without transporting MTC which is rather difficult as MTC is a toxic liquid at room condition.

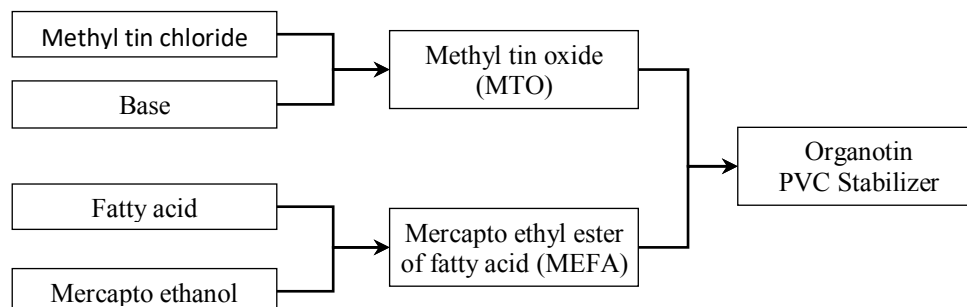


Fig. 16. The synthesis route of organotin PVC stabilizer.

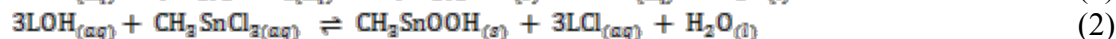
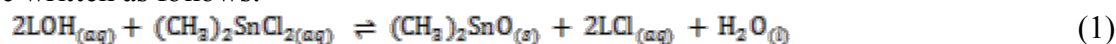
There are two methods of synthesizing MTO. The first is by reacting tin powder and primary alcohols at temperatures of 200~400°C⁵. However, this method is considered to be unattractive because it involves high reaction temperatures and requires cooling at very low temperatures to obtain MTO⁶. The second is by hydrolysis in which n-alkyl tin halides are reacted with base solutions at 60~100°C. The yields of MTO via this reaction were found in the range of 97.9~98.9%⁷. The recommended concentrations of base were between 5% and 50%⁸. The reaction is also possible be carried at lower temperature.

Although the synthesis of organotin stabilizer from MTO and MEFA has been patented in 1968, however, there is no intensive study on this topic, especially on the more downstream, i.e., reacting the obtained MTO with MEFA to produce PVC stabilizer and testing the stability effectiveness. The production of organotin stabilizer via MTO is mostly kept as proprietary know-hows by the producers. It is the purposes of this research to synthesize organotin stabilizers using MTO and MEFA. In this paper, the works focus to the synthesis of MTO in which local MTC is used as a raw material. MTO was synthesized through hydrolysis, between base and MTC. The MTC used consisted of dimethyl tin dichloride and monomethyl tin trichloride. The reaction parameters studied are the types, concentration, flowrate, and excess of bases. Two bases were used, i.e., ammonium hydroxide and sodium hydroxide. The MEAL used was prepared from rice bran oil.

2. Experimental

MTC, a mixture of monomethyl tin trichloride and dimethyl tin dichloride containing 17.8%-w Cl, was kindly supplied by PT. Timah Industri. Aqueous solution of NH₄OH containing 25%-w NH₃ was purchased from a local supplier. NaOH with 99.99%-w purity was purchased from Merck. MEAL was obtained from the previous works^{9,10,11}. The experimental set for preparing MTO is shown in Fig. 17. MTC of 200 g was first placed in the reactor. The base solution is then fed at a determined flowrate under stirring. The reaction temperature is maintained at 25~35°C by controlling the temperature of circulating water and feeding of base solution. The temperature controller stopped feeding base solution when the reaction temperature passes the maximum allowed temperature. The pH and temperature of the solution were recorded during the reaction. The reaction was stopped after all base was fed into the reactor. The solid phase was then separated using a separating funnel to obtain crude MTO. The crude MTO was then washed with water three times to obtain final products. Tin contents in products were measured using XRF.

The reaction was carried out at various base excess (0%, 5%, 10%, and 20%), base concentration (4.0, 6.6 M), and base flow rate (2, 4, 12 mL/min). Each run was performed twice. The experimental runs are presented in Table 1. As mentioned above, the used MTC consisted of monomethyl tin trichloride and dimethyl tin trichloride. The synthesis reactions can be written as follows:



The above reactions are very exothermic. This is the reason why the reaction temperature was controlled both using circulating water and by controlling the feeding rate of base.

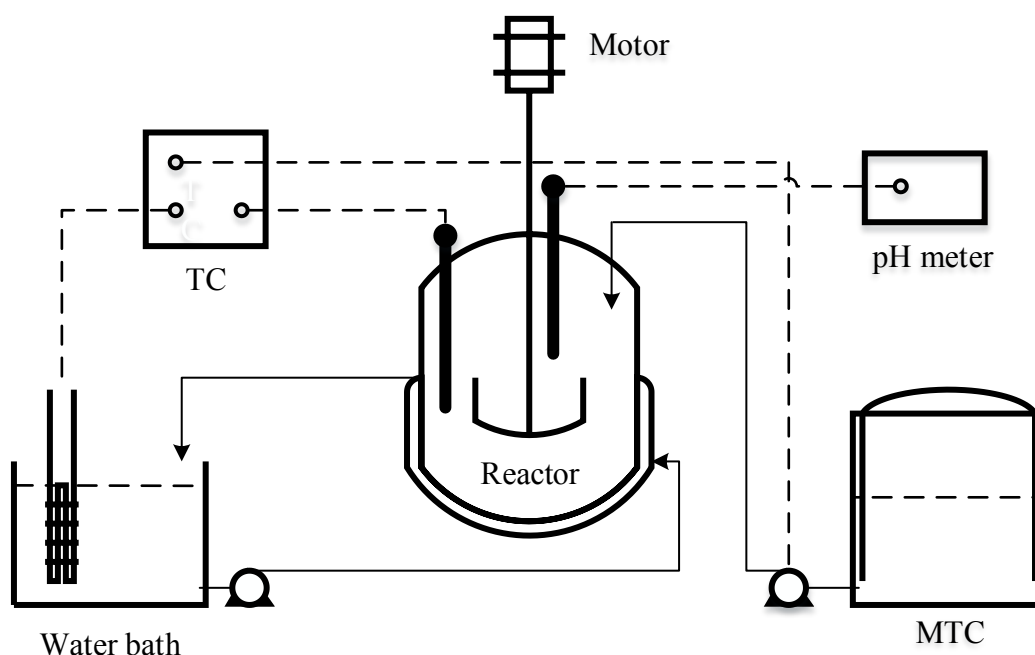


Fig. 17. Schematic of apparatus.

Table 1. Experimental runs.

Code	Base					Code	Description				
	Base	Molarity (M)	Flowrate (mL/min)	Excess (%)	Run		Base	Molarity (M)	Flowrate (mL/min)	Excess (%)	Run
SC1-1	NaOH	4.00	8.12	5	1	AF6-1	NH ₄ OH	8.79	12.19	5	1
SC1-2	NaOH	4.00	8.12	5	2	AF6-2	NH ₄ OH	8.79	12.19	5	2
SC2-1	NaOH	6.57	8.12	5	1	SE0-1	NaOH	6.57	8.12	0	1
SC2-2	NaOH	6.57	8.12	5	2	SE0-2	NaOH	6.57	8.12	0	2
SC3-1	NaOH	8.79	8.12	5	1	SE5-1	NaOH	6.57	8.12	5	1
SC3-2	NaOH	8.79	8.12	5	2	SE5-2	NaOH	6.57	8.12	5	2
AC1-1	NH ₄ OH	4.00	8.12	5	1	SE10-1	NaOH	6.57	8.12	10	1
AC1-2	NH ₄ OH	4.00	8.12	5	2	SE20-1	NaOH	6.57	8.12	20	1
AC2-1	NH ₄ OH	6.57	8.12	5	1	AE0-1	NH ₄ OH	8.79	8.12	0	1
AC2-2	NH ₄ OH	6.57	8.12	5	2	AE0-2	NH ₄ OH	8.79	8.12	0	2
AF1-1	NH ₄ OH	8.79	2.03	5	1	AE10-1	NH ₄ OH	8.79	8.12	10	1
AF1-2	NH ₄ OH	8.79	2.03	5	2	AE10-2	NH ₄ OH	8.79	8.12	10	2
AF2-1	NH ₄ OH	8.79	4.06	5	1	AE20-1	NH ₄ OH	8.79	8.12	20	1
AF2-2	NH ₄ OH	8.79	4.06	5	2	AE20-2	NH ₄ OH	8.79	8.12	20	2

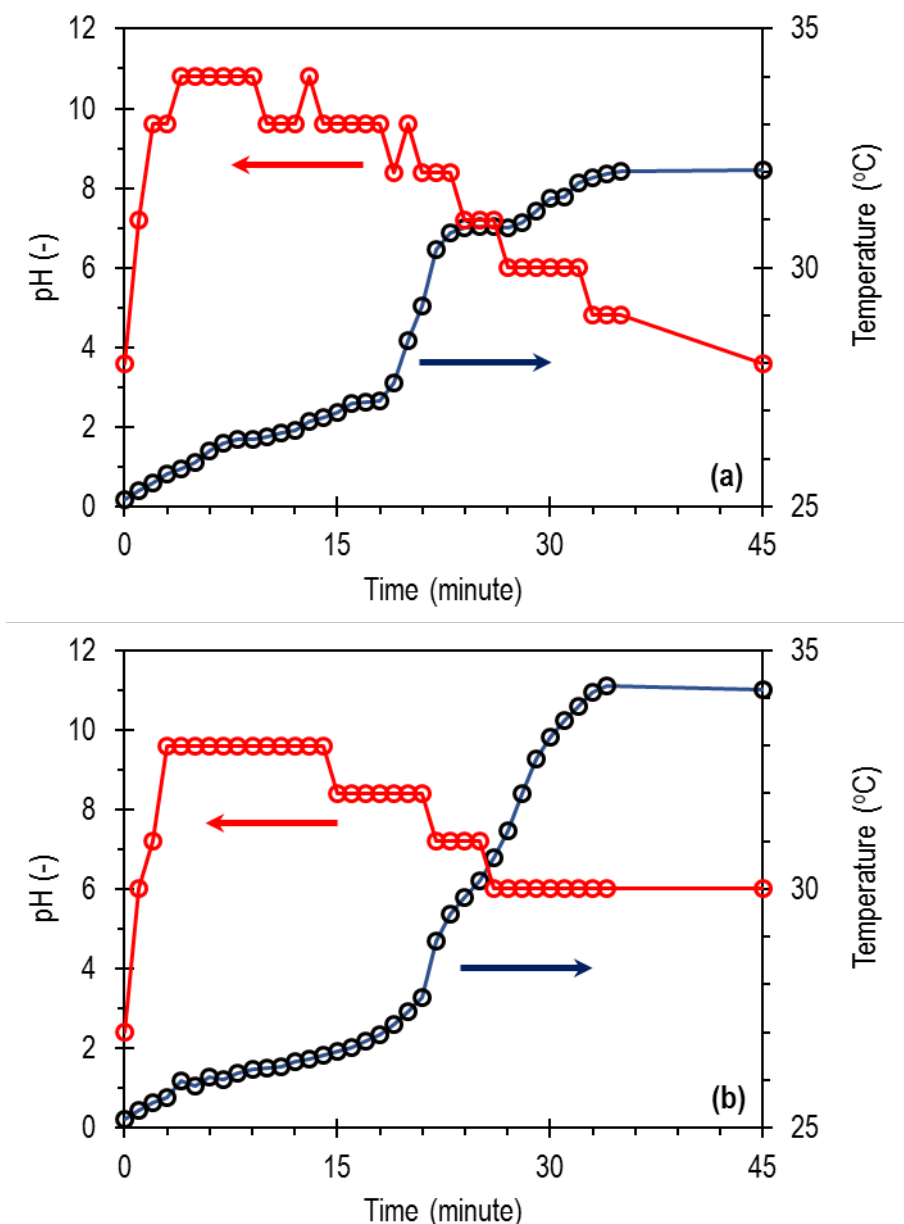


Fig. 18. Profiles of pH and temperature.

3. Results and Discussion

3.1. Profiles of pH and temperature

Fig. 18a and Fig. 18b show the pH and temperature profiles when ammonium hydroxide and sodium hydroxide were used as bases, respectively. The profiles were obtained at 4M base concentration and 5% base excess. The reaction temperature increased just after MTC was fed. It was due to the exothermic reaction occurred. After the reaction achieved a maximum point, the temperature decreased slowly as the liberated heat was absorbed by the solid phase and circulating water. When ammonium hydroxide was used, the reaction temperature could return to room temperature in 45 minutes. For sodium hydroxide, however, the temperature was still at 30°C in 45th minute. This indicate that, as sodium hydroxide is stronger than ammonium hydroxide, it gives higher reaction heat that the circulating water could not absorb the liberated heat as fast as in the case of ammonium hydroxide.

The pH of the reaction mixtures was increase continuously as the base solution was fed. The pH started at a low pH as MTC is acid. The addition of base solution neutralized part of

MTC so that pH was getting higher and higher. The equivalent points were clearly shown by the graphs. The final pH for sodium hydroxide was found to be higher than that for ammonium hydroxide. This occurred as sodium hydroxide is stronger than ammonium hydroxide.

3.2 Effect of base parameters on yield

The effects of base parameters on the yield of MTO are presented in Fig. 19a. Fig. 19a shows the effects of bases on yields both for ammonium hydroxide and sodium hydroxide. Lower yield was obtained for sodium hydroxide. This means that stronger base provides lower MTO yield. For the same excess, stronger base gave higher final pH. The higher the pH, the more soluble the MTO¹¹. Higher solubility resulted in higher loss during washing and filtration. More MTO was carried away in the filtrate so that the yield decreased.

Fig. 19a also shows the effects of base concentrations on the obtained methyl tin oxide. Higher base concentrations result in greater yield. This is because the synthesis reaction of MTO is an equilibrium reaction. The addition of base concentration causes the system to shift toward MTO to minimize the impact of disturbance according to Le Chatelier principle.

Fig. 19b shows the effects of base flow rates on yield. The effect of base flow rate is observed by comparing runs AF1-1, AF1-2, AF2-1, AF2-2, AF6-1, and AF6-2. The data in Fig. 19b shows the effect of the base flow rate on the yield of methyl tin oxide product by 0-0.4%. The value of the change given is so small that the effect of the base flow rate on the yield of methyl tin oxide product can be neglected. This means that the reaction rate of methyl tin oxide formation is very fast due to a good agitation process.

The effects of base excess on yield is shown in Fig. 19c. The graphs show an optimum base excess both for ammonium hydroxide and sodium hydroxide. The best base excesses of 10% and 5% were found for ammonium hydroxide and sodium hydroxide, respectively. When base excess was increased at the lower region, the yield also increased. This happened as the increase in excess convinced the completion of reaction. However, when excessive base excesses were applied, the pH solution increased resulting in significant product losses due to solubility. These opposite effects resulted in an optimum base excess.

3.2 Tin Content

Fig. 20 shows the content of tin in product, at various base excess. The tin contents were in the range of 63-65 and 63-66 %-w for ammonium hydroxide and sodium hydroxide, respectively. The variations in tin contents were in the range of experimental errors. Thus, it could be said the tin contents were in an average value of 64%. The theoretical tin content, estimated from the stoichiometric reaction and chlorine content of MTC is 71%. From these values, the purity products obtained was around 90%. The tin contents were a little bit lower than the theoretical value. Analysis of metals in MTO of all experimental runs found that the tin contents were in the range of 98.9-99.5, as shown in Fig. 21. It means that there was no sodium or other metals left in the products. In other words, the excess sodium could be removed by washing. Thus, the fact that the tin contents were lower than the theoretical value should be caused by no metal containing side products. It is possible some products were hydrated which lowering the tin contents in the products. In contrast to yield, optimum base excesses of 10% and 5% were found for ammonium hydroxide and sodium hydroxide, respectively, for tin content. Thus, generally speaking, the optimum base excess was in the range of 5~10%.

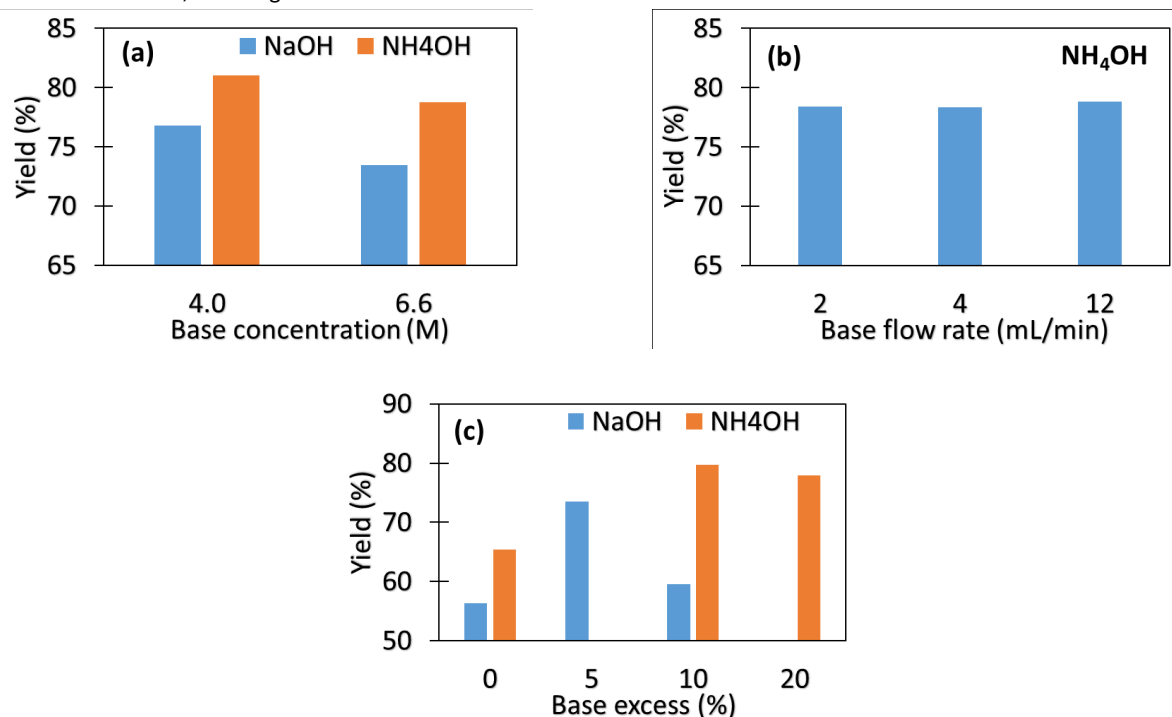


Fig. 19. Effects on yield of (a) base concentration, (b) base flow rate, and (c) base excess.

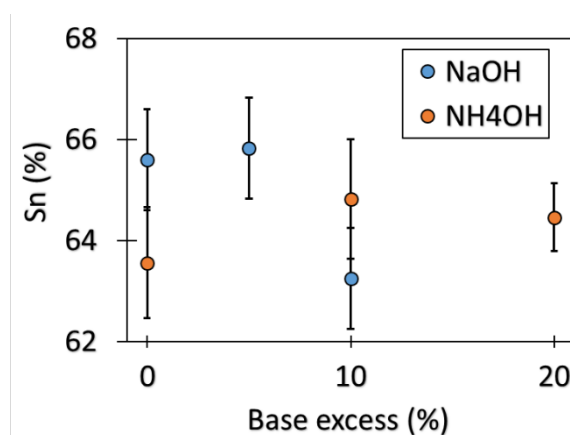


Fig. 20. Tin content of MTO, total base.

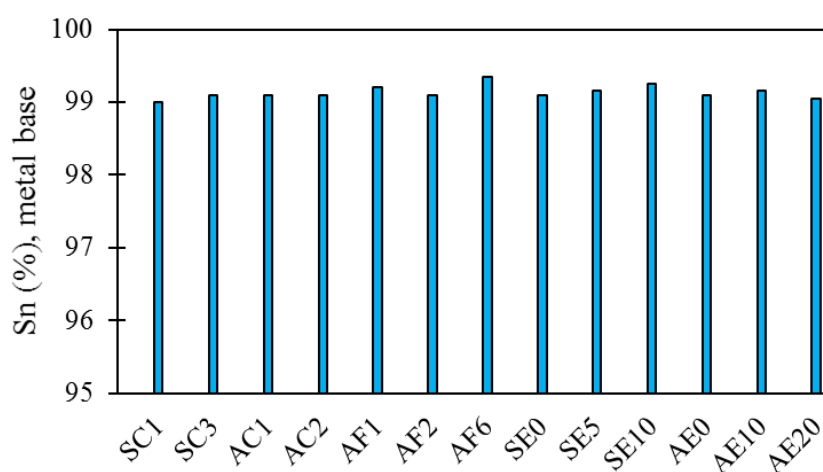
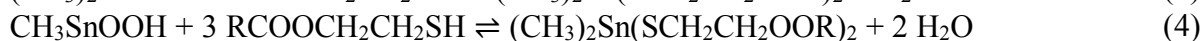
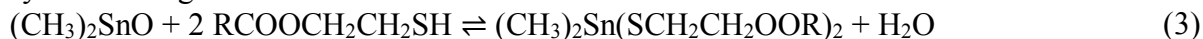


Fig. 21. Tin content of MTO (metal base).

3.3 Synthesis of stabilizer

The MTO produced was reacted with mercapto ethyl ester of rice bran fatty acid to synthesize organotin stabilizer. The reactions can be written as follows:



The reactions were carried at 80°C under rigorous stirring and vacuum. A clear oil phase was obtained in two hours of reaction. A formulation consisting of 1.5 phr the produced organotin stabilizer, 1.2 phr P-1141, 0.3 phr Loxiol G-78, 5 phr B-622, 1.5 phr PA-20, and 0.3 phr ACP 316-A was prepared. PVC resin having a k value of 57 was used. This formula is commonly applied in industry. The formulation was heated at 190°C in a two rolls mill. Samples were taken at every 5 minutes. The results are depicted in Fig. 22. Organotin stabilizer at 1.5 phr made in this research could stabilize PVC until 30 minutes. Practical extrusions require 15 minutes stability. The thermal stabilizer of this study, therefore, obeys the practical requirement. Observations during the synthesis of organotin stabilizer found that a very little solid left at the bottom of the flask. This confirms that the MTO obtained contained a small amount of impurity. Using pure MTO, no solid should be left at the end of reaction. The produced water, as a by product, evaporates due to vacuum. It is the additional advantage of using MTO route, that the synthesis of organotin stabilizer produced no waste.

















Time (min)	5	10	15	20	25	30	35	40	45
No stabilizer									
With 1 phr stabilizer									

Fig. 22. Stability test.

4. Conclusions

Methyl tin oxides have been prepared from methyl tin chloride and base solution. It was found that base flow rate did not influence the yield of methyl tin oxide. Stronger base resulted in lower yield of methyl tin oxide. The higher the base concentration, the larger the yield obtained. The optimum base excess was in the range of 5~10%. The tin content of methyl tin oxide obtained was in the range of 62% to 66%, a little bit lower than the maximum theoretical yield. The methyl tin oxide obtained was used to synthesize organotin thermal stabilizer. The thermal stabilizer has been tested using a two rolls mill and was found to be effective in stabilizing poly vinyl chloride.

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