

SYNTHESIS OF POLYMER INCLUSION MEMBRANES BASED ON PVC CONTAINING COPOLY-EDVB 4% AS A CARRIER FOR REMOVAL OF PHENOL SOLUTIONS

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ABSTRACT

Phenols are widely produced and used for industrial purposes. As a result, many phenol wastes are found that are wasted in the environment, especially the aquatic environment. Because of the nature of phenols that can harm living things, the process of separating phenols from waters was performed by the Polymer Inclusion Membrane (PIM) method. The PIM membrane was prepared with a composition of carrier compound copoly (eugenol-DVB) 4% which has been synthesized, polyvinyl chloride (PVC) as supporting compounds, and diethyl benzene (DBE) as plasticizers. The result of the synthesis of copoly-EDVB 4% produced a slightly orange colored powder with a yield of 92%, melting point in the range of 98-101 °C and soluble in organic solvents such as chloroform, tetrahydrofuran, and diethyl ether. The result of IR spectroscopy on copoly-EDVB 4% powder showed the occurrence of cross links between eugenol and divinyl benzene (DVB) shown by the presence a wave number at around 3511.2 cm⁻¹ as indication of -OH group from eugenol. The TG-DTA analysis was also carried out on Co-EDVB 4% crosslinked powder to monitor the changes in sample weights towards reference. The phenol transport test on PIM membrane with co-EDVB 4% as a carrier compound with membrane reusability analysis, with five repetitions has also been done to test the stability and durability of the membrane. The IR spectroscopy was done to characterize the PIM membranes before and after phenol transport in the repeated use and artificial waste tests. The phenol content transported from the source phase to the receiving phase was analyzed by UV-Vis spectroscopy.

KEY WORDS : Liquid membrane, Polymer inclusion membrane, Recovery, Wastewater

INTRODUCTION

The use of chemicals, especially phenols in the industrial sector both in Indonesia and throughout the world has significantly increased the impact on the environment. The extensive use of phenol and its derivatives causes by products in the form of chemical pollutants from industrial wastes that are wasted into the environment, especially in water areas. Phenol wastes discharged into the water produce unpleasant odors and are harmful to human health (Benosmane *et al.*, 2018). Phenol contained in water at a concentration of 0.001 ppm is difficult to detect using the human five senses because it has no taste and odor. Most of the phenol

pollutants that are discharged into water areas cannot be decomposed biologically, so special handling is needed in dealing with these wastes (Othman *et al.*, 2015).

Many methods have been used to remove phenols from industrial wastewater, such as adsorption using activated carbon (Beker *et al.*, 2010; Hameed and Rahman, 2008; Malusis *et al.*, 2010; Stavropoulos *et al.*, 2008; Tseng *et al.*, 2010), photocatalyst (Desrosiers *et al.*, 2006), using N-octanilpyrrolidine (Li *et al.*, 2004) and ozonation technique (Mozhdehvari *et al.*, 2009). Many researchers have tried to separate phenol by activated carbon adsorption. In addition, the phenol adsorption has also been done by the adsorbent of

amine group polymer in cyclohexane (Huang *et al.*, 2007). The phenols that have polluted the waters and become waste were then do the separation process so that later phenol compounds can be separated from the waters and re-utilized in the industrial process. The method commonly used for the recovery process is membrane technology (Kiswandono *et al.*, 2012; Kiswandono *et al.*, 2014; Kiswandono *et al.*, 2020).

Separation technology using liquid membranes such as SLM (Supported Liquid Membrane) is widely accepted as a useful technology for many industries compared to conventional separation, but in its application this membrane still has weaknesses (Nghiem *et al.*, 2006). One way to overcome the weaknesses that exist in this SLM membrane is to add a supporting polymer in the composition of the membrane making, then it is molded to form a thin, stable, and flexible film membrane. This type of membrane is known as polymer inclusion membrane (PIM). The use of PIM membrane for the separation process, has been widely carried out by researchers, both for the separation of heavy metals (Bonggotgetsakul *et al.*, 2015; Chaouqi *et al.*, 2019; Kaya *et al.*, 2016; O'Bryan *et al.*, 2017; Sellami *et al.*, 2019; Turgut *et al.*, 2017; Wang *et al.*, 2017), as well as organic compounds (Benosmane *et al.*, 2018; Kiswandono *et al.*, 2012; Ling and Mohd Suah, 2017).

PIM is a new type of liquid membrane with a composition generally made of PVC or CTA as a base polymer, and extractants commonly referred to as carriers (Almeida *et al.*, 2012). Separation with PIM membranes is one alternative method that in recent years has attracted the attention of researchers as an environmentally friendly method that supports green chemistry. This is due to the removal of large amounts of solvents, and only a small number of carriers are needed (Croft *et al.*, 2018). The transport process in the PIM membrane requires a carrier compound as one component of the formation of the PIM membrane. Various ways to improve membrane stability and permeability depend on the base polymer or by adding a crosslinking polymer to the membrane composition (Croft *et al.*, 2018).

Carrier compounds contained in the PIM membrane affect the lifetime of the membrane in transporting the target compound to the receiving phase. Compared to SLM membranes, PIM membranes have a longer life span compared to SLM membranes. This is because the mechanism of PIM membrane transport depends on the

composition of the membrane, the surface homogeneity of the membrane. This is what makes the PIM membrane has advantages compared to SLM membranes (Sellami *et al.*, 2019). The existence of a suitable carrier compound is expected to be able to interact with the target compound so that it can carry phenol from the source phase to the membrane phase. The concentration of carrier compounds used in membrane technology is generally much smaller than conventional methods that use more solvents, because of these advantages also make PIM membranes more economical with the use of appropriate carrier compounds so that the PIM membrane selectivity becomes higher (Cho *et al.*, 2018).

The requirements in choosing a carrier compound that will be used as one of the components of making PIM membranes are to have high molecular weight and to be in accordance with the target compound to be transported (Kozłowski and Walkowiak, 2002). Carrier compounds that have been used by some researchers to remove phenols with PIM membranes from waters are using calix [4] resorcinarene (Benosmane *et al.*, 2018), N, N-di (1-methylheptyl) acetamide (Meng *et al.*, 2015), copoly-EDVB which is a copolymer compound crosslinked between eugenol and divinyl benzene (DVB) has also been noted as a carrier to transport phenols with PIM membranes (Kiswandono *et al.*, 2012). Crosslink serves to increase the molecular weight of the carrier compound.

This study uses a crosslinked carrier compound from eugenol and divinylbenzene (DVB). The eugenol compound has ether, -OH, and allyl groups. The presence of the -OH group of eugenol is expected to be compatible with the target compound (phenol), then polymerized with DVB so that the molecular weight becomes larger and produces a more selective PIM membrane in transporting phenol. Copoly-eugenol divinyl benzene (co-EDVB) 4% used as a carrier compound in this study was synthesized and then tested to transport phenol from the source phase to the receiving phase by evaluating the use of PIM membranes. The use of co-EDVB as a carrier compound was carried out by (Kiswandono *et al.*, 2012) with co-EDVB concentrations of 2%, 6%, and 12% for phenol transport optimization test. Then, Kiswandono *et al.*, (2014) also continued his research on the ability of these carrier compounds to transport phenols under various conditions up to the lifetime of the membrane (Kiswandono *et al.*, 2014).

MATERIALS AND METHODS

Synthesis of co-EDVB 4%

A mass of 5.8 g Eugenol was placed into a three-neck flask and reacted with a DVB compound (divinyl benzene) in the amount of 4% by weight of eugenol and then was added 0.25 mL boron trifluoride diethyl ether, $\text{BF}_3\text{O}(\text{C}_2\text{H}_5)_2$ as a catalyst. The addition of this catalyst is done every hour while stirring with a magnetic stirrer at room temperature. The catalyst was added four times. The polymerization reaction was carried out for one night and stopped by adding 1.0 mL of methanol. The formed gel is dissolved in ether and then washed repeatedly with double distilled-water to neutral pH. The solvent is evaporated with a rotary vaporizer flask at 40 °C and the residue is stored in a desiccator. Solids or polymers formed are weighed to determine the yield and characterization.

Instrumentations

Characterization was carried out using a pH meter, FTIR (Cary 630), TGA-DTA (Seiko / AXSTAR), MP-AES (Variant), and UV-Vis spectrometers for the Analytical Chemistry and Instrumentation Laboratory of FMIPA and the Integrated Laboratory and Technology Innovation Center (LTSIT) University of Lampung.

The preparation of PIM Membrane

The PIM membrane was prepared from three main components, namely PVC as a base polymer, 4% co-EDVB carrier compound, and DBE as a plasticizer. These three components were then mixed and tetrahydrofuran (THF) added as an organic solvent until the solution homogeneously was mixed. After the solution was homogeneous, then allowed to stand for three days. The PIM membrane was characterized using FT-IR spectrophotometer before and after transport.

Phenol transport test

The phenol transport test carried out was the reusability of PIM membranes. The PIM membrane was installed between two chambers for transport, each chamber was added with 50 mL of 60 ppm phenol in the source phase and 50 mL NaOH in the receiving phase. Both chambers were stirred with a magnetic stirrer at room temperature. Every 24 hours, the transport process was stopped, and the PIM membrane was dried for 2 hours. After the membrane has been dried, the PIM membrane was

weighed and reused for the second phenol transport. The repetition was carried out until the 5th repetition. The phenol concentrations contained in the source phase and the receiving phase were analyzed using a UV-vis spectrophotometer at a maximum wavelength of 456 nm by the 4-aminoantipyrine method.

RESULTS AND DISCUSSION

Synthesis and Characterization of co-EDVB 4%

The improvement of the process of transporting the target compound from the source phase to the receiving phase depends on the suitability of the constituent components in making the membrane. Components of the carrier compound as one of the main components in the manufacture of membranes must have conformity or similarity with the target compound. The suitability of the target compound with the carrier compound can increase the effectiveness of the transport of the target compound on the PIM membrane. One of them is to polymerize the active side of the carrier compound using diene compounds, such as divinyl benzene (DVB), ethylene glycol dimethacrylate (EGDMA), and diallyl phthalate (DAF). The reactive nature of the double bonds in diene compounds makes the synthesis process possible at room temperature.

In this work, the polymerization of eugenol with DVB was performed in order to obtain a larger molecular weight. The polymer produced has a light brown powder as shown in Figure 1. The melting point was then determined and its solubility was tested in chloroform, diethyl ether, and



Fig. 1. Powder of co-EDVB 4%

tetrahydrofuran (THF). Comparison of the results of the analysis of physical and chemical properties of 4% co-EDVB powder is shown in Table 1.

Based on Table 1, the melting point of the Co-EDVB is increased with the increased of percentage of cross links between eugenol and DVB, this is predicted because of the longer crosslinking polymer bond formed. The polymerization reaction of eugenol or polieugenol with crosslinking agents will increase the molecular weight of the polymer product, so that the greater molecular weight value can affect the interaction of the membrane with the target compound. This is due to the increasing number of active sides possessed by the membrane, the presence of -OH group and the benzene ring. It will be expected that the polymer synthesis can increase the transport speed to make the transport process is faster and more efficient. The prediction structure of the polymer obtained by cross linking between eugenol and DVB is shown in Figure 2.

The results of characterization by FTIR of crosslinked powder co-EDVB 4% and eugenol before crosslinking are shown in Figure 3. While the comparisons of the main functional groups from the two compounds based on their spectra are tabulated in Table 2.

Co-EDVB powder samples were also analyzed by Thermogravimetric Analysis (TGA), to measure the change in weight of a compound as a function of temperature. The reduction in mass (decomposition) of the polymer occurs because of the thermochemical reaction obtained from the data from Differential Thermal Analysis (DTA). DTA is a measurement technique based on the difference between sample and reference material. TG analysis

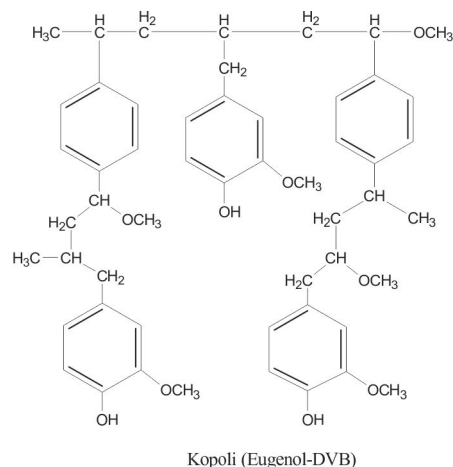


Fig. 2. The structure prediction of the crosslinked product of co-EDVB 4%

Table 1. Physical and chemical characteristic of co-EDVB powder

Sample	Rendement (%)	Colour	Melting Point (°C)	Solubility in		
				Chloroform	Diethyl ether	Tetrahydrofuran (THF)
Co-EDVB 2%*	74.16	Brown	88.4-95.7	Soluble		
Co-EDVB 4%	92.33	Brown	88.8-98	Soluble	Soluble	Soluble
Co-EDVB 6%*	71.21	Brown	89-102	Soluble		
Co-EDVB 12%*	67.42	Brown	100.6-109	Soluble		

*Reported by Kiswandono (2014)

Table 2. The comparison of the main vibration for Eugenol and co-EDVB 4%

Wave length (cm ⁻¹)	Specific group	Eugenol spectra	co-EDVB 4% spectra
817.82-842.4	Aromatic substituted group 1,2,4	Appeared in eugenol spectrum	Appeared in co-EDVB 4% spectrum
995.27-1028.7	Saturated group (vinyl, -CH=CH ₂)	Appeared in eugenol spectrum	-
1636.5650-900	Allyl group absorption (C=C stretch) C=C vibration	Appeared in eugenol spectrum	-
2929.7	The presence of CH sp ³	-	Appeared in co-EDVB 4% spectrum
3511.2	-OH group	-	Appeared in co-EDVB 4% spectrum
1602	C=C group of aromatic benzene	-	Appeared in co-EDVB 4% spectrum

was carried out in a temperature range of 30-900°C on co-EDVB with a heating speed of 10°C/min. The results of the analysis in the form of spectrum curves is shown in Figure 4.

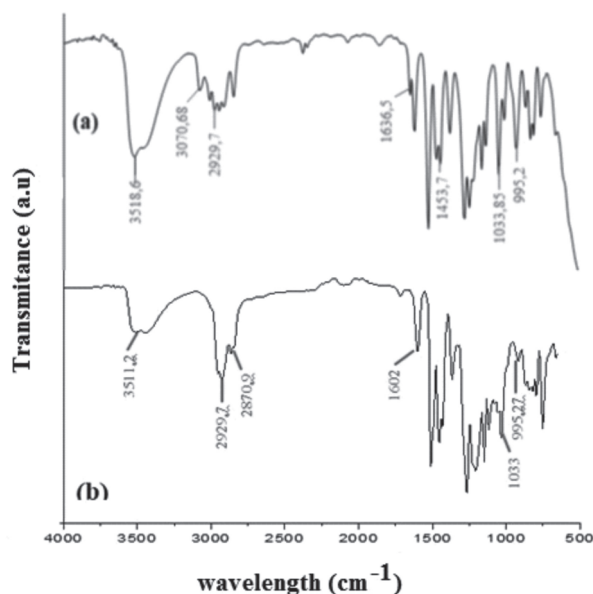


Fig. 3. The FT-IR spectra of (a) Eugenol; (b) co-EDVB 4%

The decomposition co-EDVB 4% occurs on the TGA curve with a change in weight that occurs in the temperature range 30-400.1 °C. Based on Figure 3, there are four times of weight loss. The first weight reduction occurred at a temperature of 30-105.2 °C in the amount of 2.1%, then continued with a weight reduction of 26.4% at 350.5°C. The first two weight losses are estimated related to sample dehydration, i.e. the release of water (evaporation) contained in the sample. The numbers of hydroxy groups present in the polymer estimated to affect the weight decrease is quite high.

The third weight change is a decrease of 90.5% in the temperature range 350.5-450.8°C. This weight reduction is expected due to breaking of many benzene bonds found in the polymer structure, thus requires a long time and a high temperature. The fourth weight change reached 94.9% which occurred in the temperature range of 450.8-750°C which is thought to be the loss of the CO, -CH-CH₃, and -OCH₃ groups.

The co-EDVB 4% powder as a carrier compound is then mixed together with other components namely PVC and DBE to form a PIM membrane.

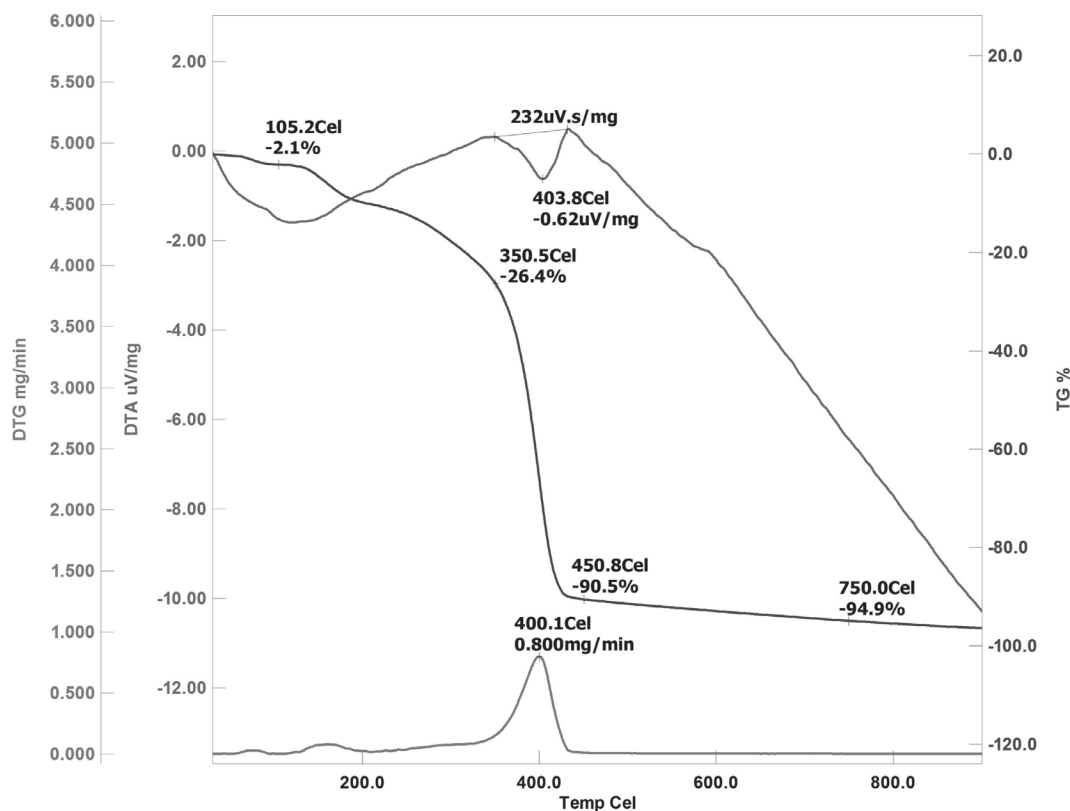


Fig. 4. Analysis curve of TG-DTA co-EDVB 4%

The PIM membrane was obtained having a clear and thin surface as shown in Figure 5.



Fig. 5. PIM membrane containing co-EDVB 4% as a carrier

The reusability PIM membrane with Phenol Transport

The stability and durability of a membrane can be tested from its reusability. The procedure for reusability of the PIM membrane was carried out five times by repeating the phenol transport for 24 hours. The result showed that in each repetition, the phenol transported to the receiving phase decreases. This is in agreement with the data in the literature (Kiswandono *et al.*, 2012; Kiswandono *et al.*, 2020). The results also indicated that on the second and third transport process, the phenol transport from the source phase to the receiving phase have decreased (Figure 6). This occurred as in the first membrane usage, there are membrane components that have leached from the carrier group in co-EDVB 4%, thus the phenol content which has been transported from the source phase to the receiving phase decreases in the second and third repetitions.

Physically it can be observed the colour changes in the receiving phase, on the first repetition of the colour of the solution in the receiving phase is more concentrated than in the second and third

repetitions. Even on the fourth and fifth repetitions, the colour of the solution becomes clearer and no change in colour. After the second and third repeated use indicates that the number of active groups in the carrier compound has significantly decreased. The decrease of active groups available in benzene rings and -OH groups causes the weaker interaction of π - π bonds and hydrogen bonds formed between phenols and co-EDVB 4%, thus decreasing the phenol transport to the receiving phase. Percentage loss of membrane components is obtained by comparing membrane weight after use with before use.

Figure 7 shows the curve of the effect of phenol transport on repeated membrane usage, it can be seen that the more membrane is reused, the membrane's ability to transport the phenol decreases significantly as evidenced by the reduction in percent of phenol obtained in the receiving phase. On the 4th and 5th repetitions of the PIM membrane, very less phenol was transported from the source phase to the receiving phase, as the amount of phenol transported is so small or it is

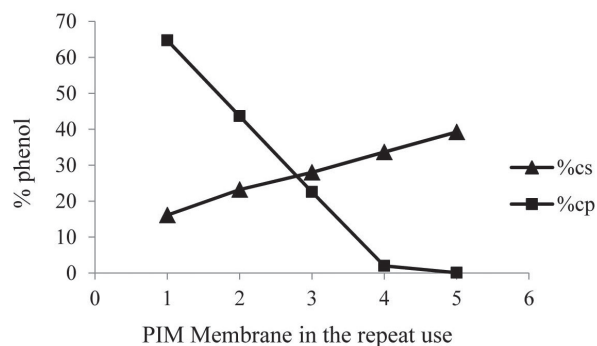


Fig. 7. The graph of the effect of phenol transport to the reusability of PIM membrane

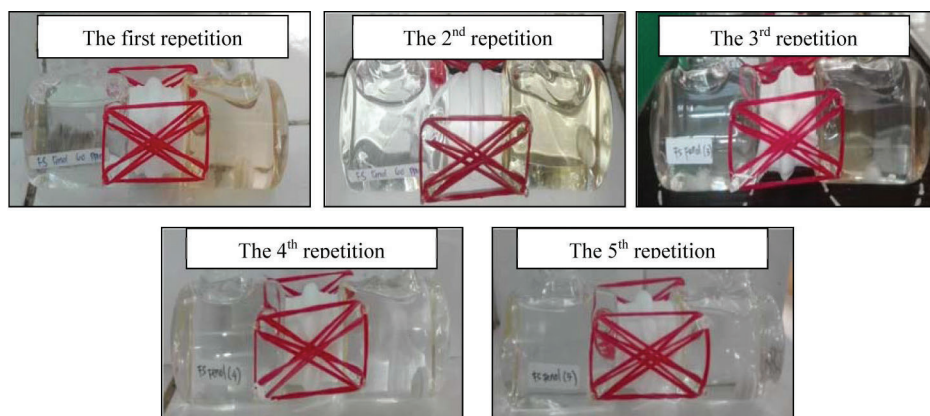


Fig. 6. The colour changes occurred during the phenol transport on reusability test of PIM membrane

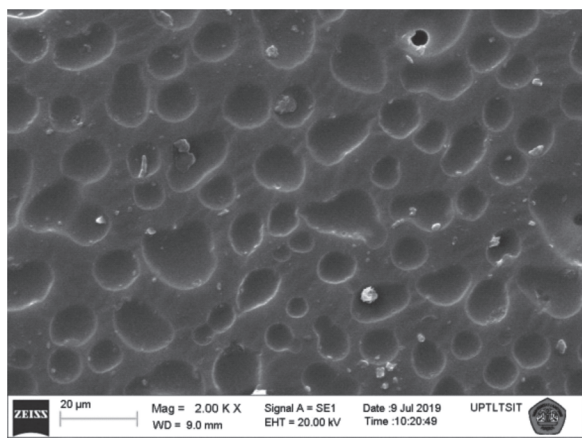


Fig. 8. The Morphology of PIM membrane after phenol transport on 5th repetition (magnified 2000x)

hardly able to transport the phenol, therefore the PIM membrane is effectively to transport phenols until the 3rd repetition.

The PIM membrane after transport is then characterized by SEM to monitor the morphology of the membrane surface after the fifth repetition. The results of SEM is shown in Figure 8. Based on this figure, the membrane has large pores and they are almost uniform indicating that the interaction has occurred between phenols and carrier compounds contained in the membrane.

CONCLUSION

Synthesis of copoly eugenol-divinyl benzene (co-EDVB) 4% has been successfully prepared and was well characterized by FTIR to see the differences of functional groups before crosslinking and after crosslinking between eugenol and DVB which characterized by the loss of allyl groups and vinyl groups at wave numbers of 1636 cm⁻¹ and 995 cm⁻¹, respectively. The phenol transport test with membrane composition of PVC as a base polymer, dibenzyl ether (DBE) as a plasticizer, and co-EDVB 4% as a carrier compound, the membrane works well and gives effective phenol transport up to the 3rd use.

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