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To cite this article: Apiched Savasdipol *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **559** 012018

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The Recovery Process of Used Hydrocarbon Solvent by Vacuum Distillation

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Abstract. In polymer production industry, a large amount of hydrocarbon solvent (HC solvent) are used to clean the reactor. Normally, used HC solvent are managed by Incineration process at high temperature (around 700 – 1200 °C) which cause a large amount of waste gas explosion such as CO₂. To manage the used solvent for energy saving and global warming reason, used HC solvent should be recycled by refining process. The aliphatic hydrocarbon, EXXSOL D80, was selected as the model HC solvent in this research work. The vacuum distillation was selected as the suitable refining process. To determine the optimum refining condition, the effect of vacuum distillation pressure (13, 15, 20, 25 and 30 kPa) on % yield, purity and assessment of economic value was studied. The purity of used solvent and %carbon atom content were confirmed by gas chromatography-mass spectrometer (GC-MS). %Purity of carbon-12, 13 and 14 atom was decreased with increasing distillation pressure. The economic cost was evaluated. At The suitable condition, 170 °C 13 kPa, the highest yield of distilled solvent was observed at 77.18%. Thus the recovery used solvent can be reduce cost for purchase and disposal around 33,651.27 USD/year. Finally, vacuum distillation is suitable for recovery used HC solvent.

1. Introduction

The growth of polymer production business is rapidly expanding because polymer products were consumed in daily life. A lot of chemical substances were used as curing agent, color, catalyst, retardant, solvent. Cleaning equipment process is very important process in polymerization production. HC solvent was frequently used in cleaning process for mixer and container.

Aliphatic HC solvent is an organic compound. HC solvents consists of C₁₂ – C₁₄ saturated hydrocarbons. The physical properties, boiling point, specific gravity (at 15.6 °C) and vapour pressure (at 20 °C) of HC solvent is 209 – 228 °C, 0.80 and 0.2 mmHg. The flash point is 76.7 °C and auto-ignition temperature is 216 °C [1].

In this research, the recovery process of used HC solvent was studied by distillation process. Vacuum distillation was interested for recovery used HC solvent because vacuum distillation can reduce pressure to very low, thus the rate of distillation is increased [2] and the equipment can be easily set up. The influence of distillation pressure on recovery used HC solvent for purity and efficiency was investigated. The chemical and economical evaluation was also interested. The appropriate condition of vacuum distillation will be discussed for the new selection of used HC solvent recovery process.



2. Methodology

2.1 Materials

Reference HC solvent, EXXSOL D80, Dearomatized Fluids, (Union Petrochemical Public Company Limited) were used. Used HC solvent were as received from silicone rubber production cleaning process. The residue silicone oligomer and impurities from used HC solvent were firstly removed by filter (Nylon Monofilament Mesh (NMO) sized of 80 μm).

2.2 Vacuum distillation process

The vacuum distillation conditions were corresponded to Cox's chart vapour pressure [3]. N-dodecane ($\text{C}_{12}\text{H}_{26}$) was selected as reference solvent for the identification standard parameter in these experiment. The distillation temperature and pressure was 170 $^{\circ}\text{C}$ and 13 kPa, respectively. To investigate the effect of distillation pressure, various distillation pressure (13, 15, 20, 25 and 30 kPa) were used. The schematic diagram of the recovery process used HC solvent was shown in figure. 1. The experimental set up was shown in figure. 2

The used HC solvent of 250 ml was poured into round bottom flask (size of 500 mL). The used HC solvent were heated at temperature of 170 $^{\circ}\text{C}$ and at distillation pressure of 13, 15, 20, 25 and 30 kPa. The vacuum distillation conditions were tabulated in table 1.

Table 1. The vacuum distillation conditions.

Conditions	Details	Conditions	Details
170 $^{\circ}\text{C}$, 13 kPa	1) Step 1: 30 $^{\circ}\text{C}$, 70 kPa, at start. 2) Step 2: 60 $^{\circ}\text{C}$, 50 kPa, at 20 min. 3) Step 3: 90 $^{\circ}\text{C}$, 30 kPa, at 48 min. 4) Step 4: 110 $^{\circ}\text{C}$, 15 kPa, at 67 min. 5) Step 5: 130 $^{\circ}\text{C}$, 13 kPa, at 85 min. 6) Step 6: 170 $^{\circ}\text{C}$, 13 kPa, for 5 hours.	170 $^{\circ}\text{C}$, 15 kPa	1) Step 1: 30 $^{\circ}\text{C}$, 70 kPa, at start. 2) Step 2: 60 $^{\circ}\text{C}$, 50 kPa, at 20 min. 3) Step 3: 90 $^{\circ}\text{C}$, 30 kPa, at 49 min. 4) Step 4: 110 $^{\circ}\text{C}$, 15 kPa, at 79 min. 5) Step 5: 170 $^{\circ}\text{C}$, 15 kPa, for 5 hours.
170 $^{\circ}\text{C}$, 20 kPa	1) Step 1: 30 $^{\circ}\text{C}$, 70 kPa, at start. 2) Step 2: 60 $^{\circ}\text{C}$, 50 kPa, at 20 min. 3) Step 3: 90 $^{\circ}\text{C}$, 30 kPa, at 48 min. 4) Step 4: 110 $^{\circ}\text{C}$, 20 kPa, at 66 min. 5) Step 5: 170 $^{\circ}\text{C}$, 20 kPa, for 5 hours.	170 $^{\circ}\text{C}$, 25 kPa	1) Step 1: 30 $^{\circ}\text{C}$, 70 kPa, at start. 2) Step 2: 60 $^{\circ}\text{C}$, 50 kPa, at 20 min. 3) Step 3: 90 $^{\circ}\text{C}$, 30 kPa, at 50 min. 4) Step 4: 110 $^{\circ}\text{C}$, 25 kPa, at 70 min. 5) Step 5: 170 $^{\circ}\text{C}$, 25 kPa, for 5 hours.
170 $^{\circ}\text{C}$, 30 kPa	1) Step 1: 30 $^{\circ}\text{C}$, 70 kPa, at start. 2) Step 2: 60 $^{\circ}\text{C}$, 50 kPa, at 18 min. 3) Step 3: 90 $^{\circ}\text{C}$, 30 kPa, at 36 min. 4) Step 4: 170 $^{\circ}\text{C}$, 30 kPa, for 5 hours.		

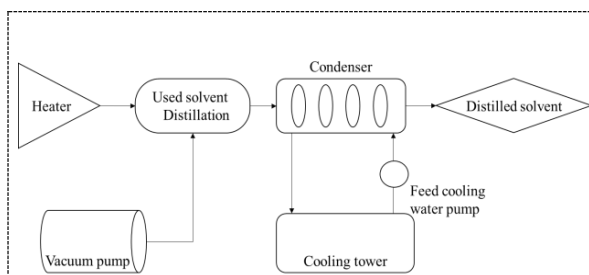


Figure 1. Schematic diagram of recovery used solvent process.



Figure 2. The experimental of vacuum distillation.

2.3 Characterization

To confirm the successful of recovery used solvent process, the purity of distilled solvent was determined by Gas chromatography-mass spectrometer (GC-MS, 7890B-GC & 5977A series GC/MSD, Agilent). Distilled solvent of 0.05 %v/v was used in GC-MS test. The condition of GC-MS is shown in table 2. The functional groups of distilled solvent was characterized by Fourier Transform Infrared Spectroscopy (FT-IR spectroscopy, Perkin Elmer).

Table 2. The condition of GC-MS characterization.

Condition	Parameter	Condition	Parameter
Type of column	HP-5 MS Ultra Inert	Carrier gas	Helium (He)
Column Oven Temp.	75.0 °C	Pressure	100.2 kPa
Hold time (1)	0.5 min	Total flow	161.5 mL/min
Injection temp	250.0 °C	Column flow	1.5 mL/min
Transfer line temp	280.0 °C	Purge flow	10.0 mL/min
Injection volume	1 mL	Split ratio	100.0
Injection mode	Split		

3. Result and discussion

3.1 Effect of pressure on %yield recovery used HC solvent process

The effect of temperature on %yield of recovery used HC solvent was studied from our previous work [4]. The experiments were conducted at pressure of 13 kPa and temperature of 140, 150, 160 and 170 °C. The % yield increased with increasing vacuum distillation temperature due to increasing energy for breaking the bond. The maximum %yield was obtained from vacuum distillation temperature of 170 °C. At higher than 170 °C, the bumping was observed. When the vacuum distillation temperature increased, the %yield of recovery used HC solvent increased corresponding to increasing of energy consumption too [4]. Thus turning the economic worthiness condition is required. To investigate the suitable condition for used solvent waste recovery process, the vacuum distillation process was conducted at 170 °C and pressure was varied at 13, 15, 20, 25 and 30 kPa. The %yield of recovery used HC solvent was determined and shown in figure. 3. The % yield decrease with increasing pressure which corresponding to relation between pressure and boiling point of substance. When pressure reduced to very low pressure as the result of the low vapour density was occurred, thus the superficial vapour velocity increased. Due to the effect of pressure on %yield, it is possible to reduce time for distillation [5].

3.2 Distilled solvent characterization

To confirm the functional group of distilled solvent from recovery of used HC solvent process by vacuum distillation. The functional group of distilled HC solvent and reference solvent were first

investigated by FT-IR spectroscopy (Figure. 4). The distilled HC solvent shows the similar peak as reference. Thus the distilled HC solvent was successfully recovered by vacuum distillation process.

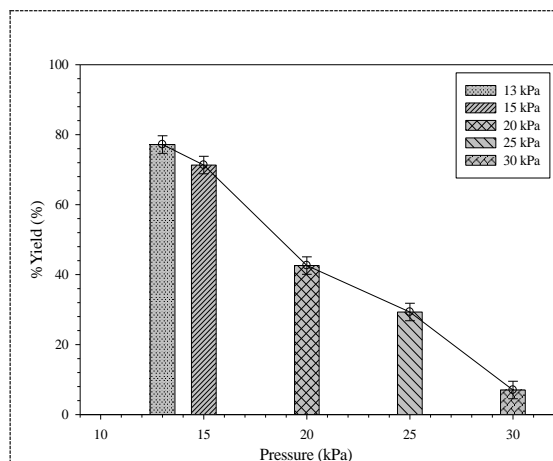


Figure 3. % Yield of distilled solvent at various pressures in vacuum distillation.

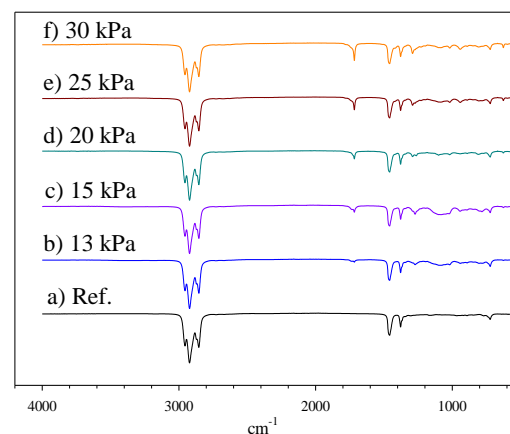


Figure 4. The functional group of a) reference solvent and distilled solvent b) at 13 kPa, c) at 15 kPa, d) at 20 kPa, e) at 25 kPa and f) at 30 kPa.

The important functional group of aliphatic hydrocarbon 2954.98 , 2921.89 and 2853.88 cm^{-1} for C-H stretching of alkane, 1457.78 cm^{-1} for C-C stretching of aromatics, 1377.87 cm^{-1} for C-H bending of alkane and 723.09 cm^{-1} for C-H bending of alkene, respectively [6]. From FT-IR spectrum, the peak at 1710.53 cm^{-1} for C=O stretching of carbonyl was observed which corresponding to silicone elastomer production about the one component silicone rubbers, acetoxy silicone. Their designations refer to the by-products generated during the curing process [7]. The structure acetic acid is -COOH and this structure has carbonyl group. High pressure as the result of relative volatility and flow rate of distilled solvent between the components will become smaller compared with low pressures [8]. The GC-MS results might be clarified-these evidences.

3.3 Purity of distilled solvent

To evaluate about %carbon atom content and purity of distilled solvent, the distilled solvent was characterized by GC-MS. Total Ion Chromatogram (TIC) of distilled solvent was investigated and shown in figure. 5 and %carbon atom content of C_{11} , C_{12} , C_{13} and C_{14} is shown in table 3.

From table.3 the %content of C_{12} , C_{13} and C_{14} in distilled HC solvent were characterized by GC-MS.

The %carbon atom content of C_{12} , C_{13} and C_{14} under 170°C 13 kPa condition were found highest carbon content when compared with distilled HC solvent at 15, 20, 25 and 30 kPa which corresponding to substance of low molecular weight can be satisfactorily distilled under low pressure provided its high concentration low molecular weight hydrocarbon content [9]. The %purity of distilled HC solvent is shown in table 4.

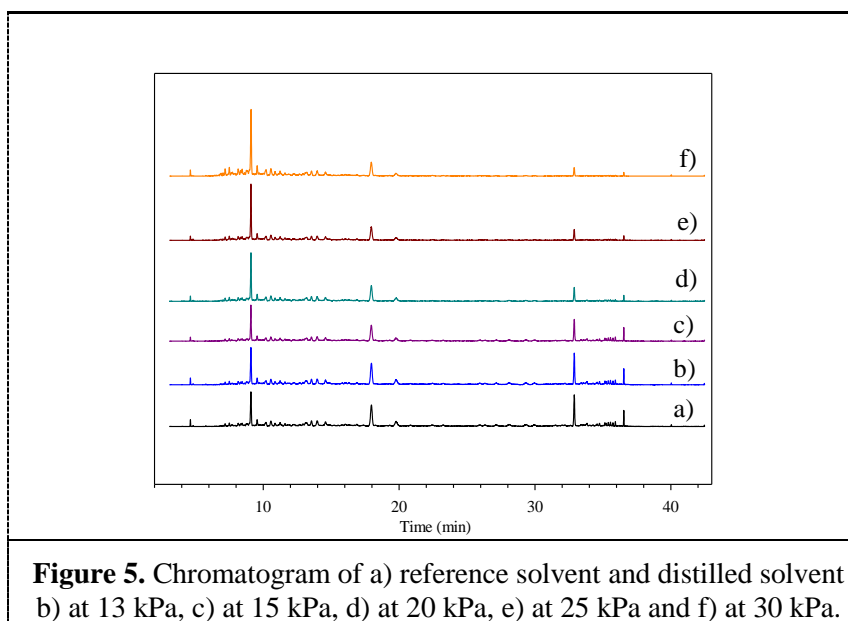


Table 3. The %carbon content of C₁₂, C₁₃ and C₁₄ in distilled HC solvent.

Solvent	%Carbon atom content (%)		
	C12	C13	C14
EXXSOL D80	35.46	30.52	10.86
Distillation pressure 13 kPa	34.93	27.06	6.02
Distillation pressure 15 kPa	34.18	25.88	5.21
Distillation pressure 20 kPa	34.66	16.71	3.23
Distillation pressure 25 kPa	30.84	16.92	3.24
Distillation pressure 30 kPa	25.03	8.28	1.59

From table. 4 the purity of C₁₂, C₁₃ and C₁₄ in distilled HC solvent were characterized and calculated. The % purity of distilled HC solvents were compared with reference solvent. From the result at 170 °C 13 kPa condition was found C₁₂, C₁₃ and C₁₄ higher than 15, 20, 25 and 30 kPa, respectively. Because high molecular weight as C₁₃ and C₁₄ was produced from liquid phase to vapour phase less than low molecular weight as C₁₂ due to high pressure was affected to vapour density. As the result of %purity of C₁₂ at 13, 15, 20, 25 and 30 kPa were increased with increasing distillation pressure but in C₁₃ and C₁₄ were decreased with increasing pressure [10].

Table 4. The %purity of distilled HC solvent.

Condition	%Purity (%)		
	C12	C13	C14
Distillation pressure 13 kPa	98.51	88.66	55.43
Distillation pressure 15 kPa	96.39	84.80	47.97
Distillation pressure 20 kPa	97.74	54.75	29.74
Distillation pressure 25 kPa	86.97	55.44	29.83
Distillation pressure 30 kPa	70.59	27.13	14.64

3.4 Economic evaluation of distilled solvent

To study the economic evaluation of distilled HC solvent, the working day is 320 day/year. 200 L of HC solvent was used for cleaning process per day. The cost for purchased HC solvent is 120.76 USD/200L and cost for disposal used HC solvent per year is 4,954.24 USD. Total of cost for purchase and treatment is 43,597.31 USD/year.

The condition 170 °C and 13 kPa in recovery process was evaluated. 77.18% of yield was obtained to highest yield of distilled HC solvent. From the result of chemical properties and %carbon atom content is similar with reference HC solvent cause of this condition is suitable for refining process.

Total of recovery solvent per year can be distilled 49,395 L/year. Cost reduction for disposal per year can be reduce to 3,823.65 USD/year. Cost reduction for purchase new HC solvent can be decrease around 29,827.62 USD/year.

4. Conclusion

The suitable condition of recovery used HC solvent by vacuum distillation was studied. The %yield of distilled HC solvents were found in 170 °C 13 kPa condition was produced highest yield at 77.18% of recovery process. The chemical composition of distilled solvents were characterized by FT-IR. The distilled solvent shows the similar peak as reference. But at 1710.53 cm⁻¹ was found carbonyl composition when distillation pressure was increased at 15, 20, 25 and 30 kPa which corresponding to silicone elastomer production about the one component silicone rubbers, acetoxysilicone because the curing agent was used acetic acid (-COOH). The %carbon atom content and purity of distilled solvent were determined by GC-MS. The carbon content 34.93% and purity 98.51% of C₁₂ at 170 °C 13 kPa was obtained higher than 15, 20, 25 and 30 kPa. But the carbon content and purity of C₁₃ and C₁₄ were reduced with increasing distillation pressure due to distillation pressure was affected to vapour density. When distillation was used low pressure as the result of low vapour density substance was occurred and effected to high molecular weight (high vapour density) was not occurred. Thus the recovery used HC solvent process will be reduce amount of used solvent for disposal around 49,000 L/year. The cost reduction was evaluated and the cost can be reduced 29,827.62 USD/year. Finally, vacuum distillation is suitable for recovery used HC solvent.

5. References

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Acknowledgments

The authors would like to thanks the financial support from Smart (2015) Services limited and National Science Technology and Innovation Policy Office for support this research.