The Experimental Research on the Regeneration and Recycling Feasibility of the Waste Heat Transfer Fluids

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Abstract. The regeneration recycling feasibility of the waste heat transfer fluids (HTF) of seven companies in Guangzhou were researched using the rotary steam-filtration-adsorption method, respectively, and the indicators of reclaimed oil were tested according to the current standards. The experimental results showed that the rotary steaming-adsorption-filter regeneration method had the advantages of simple operation, low energy consumption, low processing cost. The good oil in waste heat transfer fluids was about 50% through the regeneration recycle. This experimental method can be used for regeneration recycle of three companies.

Introduction

As a kind of heat transfer media, HTF has been widely used in chemical industry, printing and dyeing, rubber, resin, paint, road construction, paper making, and food industry[1-2] almost various occasions in recent years[3-5] for more and more usages and dosage[6-11]. HTF is mostly composed of hydrocarbons[12] and that is easy for a thermal cracking, oxidative condensation or any raw coke chemical changes, resulting in a decline in oil performance or even a scrap[13-14]. Accounting for most part of the waste oil is the function component and it has the value of recycling. Though the waste heat conduction oil would be used as a fuel oil supply for the refinery, it is a serious waste of resources, moreover, it will lead to an environmental pollution[15-16] for which now there having regulations and management system standards[17]. Therefore, to regenerate and recycle the waste HTF has very important practical significance for the enterprises to reduce the production cost, to save energy and protect the environment [18].

In chemical industry, a precipitation, the sulfuric acid refining, a clay treatment and a filtration are complex and are difficult to operate for HTF regeneration as the main procedures, besides, the high investment cost and secondary pollution from the improper disposal of regenerated waste liquor [19-21], making it hard to be accepted by people. In this paper, waste conduction oil from seven companies in Guangzhou was taken as the research object. In the regeneration process, procedures as reduced pressure rotary steaming, adsorption and filtration were adopted to deal with the oil from seven enterprises with comparative study, respectively. Then every index of the oil was analyzed according to current standards to determine the feasibility of the renewable technology based on the experimental results.

Methodology

Regeneration Experiment Steps

Weigh 80~90g waste oil into the bottom of the flask and use the rotary evaporator to remove moisture and low boiling point of organic compounds until no liquid flowing under the condition that the water bath temperature is 70 °C and the vacuum degree is 0.1 MPa. Weigh the silicone as a filter aid at 25~30% the weight of the waste oil, place the silica gel in the glass sand core hopper
and smooth the compaction gently. Connect the suction filter device and then pour the sample processed by the rotary evaporator into the funnel for a suction filter to remove carbon residue and insolubles.

**Reclaimed Oil Recovery Method**

The recovery rate of the regenerated oil is given by the formula:

\[ \eta = \frac{m_1}{m_0} \times 100\% \tag{1} \]

where, \( m_1 \) and \( m_2 \) are weight of the crude oil and weight of the regenerated oil, respectively.

**Detection Method for Indicators of Regenerated Oil**

The detection methods and technology requirements for regenerated oil were in accordance with provisions about organic heat carriers as in ISO14001 and ISO17025[17,22] and others as follows.

<table>
<thead>
<tr>
<th>Items index of standard</th>
<th>Test method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Open flash point(℃)</td>
<td>&gt; 180 ISO2592-2000</td>
</tr>
<tr>
<td>Kinematic viscosity(40℃)/(mm²/s)</td>
<td>( \leq 40 ) ISO3104:1994</td>
</tr>
<tr>
<td>Carbon residue %(m/m)</td>
<td>( \leq 0.05 ) ISO10370-1996</td>
</tr>
<tr>
<td>Acid value/(KOH mg/g)</td>
<td>( \leq 0.05 ) ISO6618:1997</td>
</tr>
<tr>
<td>Moisture content (mg/kg)</td>
<td>( &lt; 500 ) ISO760:1978</td>
</tr>
</tbody>
</table>

Flash point is an indication of the ability of a substance to form a flammable mixture with air under controlled conditions, and then to support combustion, it is applicable to petroleum products having an \( T_f \) (open flash point) above 79℃, except fuel oils, which are most commonly tested by the closed cup procedures described in ISO2719[28]. The open flash point is a number of property that may contribute towards the assessment of overall flammability and combustibility of a material.

The Kinematic viscosity of the sample could be calculated by the following relation:

\[ V_t = C \times \tau \tag{2} \]

where, \( C \) the calibration constant of the viscometer, \( \tau \) the mean float time.

The carbon residue value serves as an approximation of the tendency of petroleum products to form carbonaceous deposits under similar degradation conditions, and can be useful in the assessment of relative carbon-forming tendencies of products within the same class. Calculate the mass percentage of carbon residue in the sample, using equation (3).

\[ X = \frac{m_4-m_2}{m_3-m_2} \times 100 \tag{3} \]

\( X \) refers to the carbon residue(m/m), \( m_2, m_3 \) and \( m_4 \) represent the mass of” the empty vial”, “vial + test portion”, “vial + residue”, respectively. According to (3), the result must be a percentage.

Quantity of base, expressed in milligrams of potassium hydroxide (KOH) per gram of sample that is required to titrate the acid constituents present in 1 g of sample when titrated under prescribed conditions. The acid number, AN, in milligrams of KOH per gram of the test sample, from the equation:

\[ AN = \frac{(V_1-V_0) \times 56.1}{W} \tag{4} \]

\( AN \) expresses acid value (KOH mg/g), and \( V_1 \) is the volume, in millilitres, of potassium hydroxide solution required for titration of the test portion, \( V_0 \) is that of the blank solution.

\[ W = \frac{T \times V_2}{m_0 \times 10} \tag{5} \]
\[ W = \frac{T \cdot V_2}{v_0 \cdot \rho \cdot 10} \]  

(6)

\( W \) refers to water content of the sample, expressed as a percentage by mass, \( m_0 \) is the mass, in grams, of the test portion (in the case of solid products), \( v_0 \) is the volume, in milliliters, of the test portion (in the case of the liquid products), \( \rho \) is the density of the sample, in grams per milliliter at 20\(^\circ\)C (in the case of the liquid products), \( v_2 \) is the volume, in milliliters, of the Karl Fischer reagent used for the determination, and \( T \) is the water equivalent, in milligrams per milliliter, of the Karl Fischer reagent.

Results and Discussion

Regenerated Oil Recovery Rate Determination Results

The average recovery rate of the regenerated oils were as in Table 2.

<table>
<thead>
<tr>
<th>Manufacturer No.</th>
<th>( W ) (mg/kg)</th>
<th>( V_t ) (40(^\circ)C)/s</th>
<th>( X ) / % (m/m)</th>
<th>AN / KOH mg/g</th>
<th>Average recovery rate %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>157.8</td>
<td>27.51</td>
<td>0.01</td>
<td>0.017</td>
<td>76.56</td>
</tr>
<tr>
<td>2</td>
<td>200.4</td>
<td>25.16</td>
<td>1.53</td>
<td>0.595</td>
<td>50.99</td>
</tr>
<tr>
<td>3</td>
<td>147.9</td>
<td>29.34</td>
<td>0.01</td>
<td>0.013</td>
<td>72.41</td>
</tr>
<tr>
<td>4</td>
<td>186.8</td>
<td>43.64</td>
<td>0.01</td>
<td>0.069</td>
<td>53.66</td>
</tr>
<tr>
<td>5</td>
<td>176.6</td>
<td>30.50</td>
<td>0.01</td>
<td>0.016</td>
<td>72.17</td>
</tr>
<tr>
<td>6</td>
<td>155.0</td>
<td>57.87</td>
<td>0.12</td>
<td>0.051</td>
<td>60.09</td>
</tr>
<tr>
<td>7</td>
<td>208.3</td>
<td>39.35</td>
<td>0.67</td>
<td>0.058</td>
<td>68.34</td>
</tr>
</tbody>
</table>

From Table 2 above, the lowest recovery rate of the regenerated oil was 50.99%, while the highest was 76.56%, describing that there existing quite an amount of good oil or qualified content in the waste HTF, deserving to be recycled.

Test Results of Kinematic Viscosity

The test results of kinematic viscosity were given in Table 2, apart from manufacturer 4 and 6, all regenerated oil from other manufacturers can reach the technical standards, showing a remarkable results of the procedures in improving kinematic viscosity of regenerated oil.

Carbon Residue Determination Results

The determination results of carbon residue were as shown in the above, from Table 2, all manufacturers could achieve the technology standard on acid value of the regenerated oil except for No.2, No.6 and No.7, approving that the regenerated method was talented for recovering the indicator of carbon residue.

Acid Value Determination Results

Test results of acid value were in Table 2, from which we knew manufacturer No.1, No.3 and No.5 could conform to the requirements of the technology standard on acid value after the regenerated process, indicating a common effect on renewing the index of acid value.

Moisture Determination Results

Moisture determination results of regenerated oil sample were shown in Table 2, with the procedures dealing with all the samples from the seven companies, the technology standard on moisture had been satisfied, it can be seen that, the procedures performed well in restoring this index.

Summary

The procedures adopted in the paper had advantages of low energy consumption, low processing cost and easy operation and it could be seen that the about 50% of the regenerated waste heat conduction oil were qualified, which could reduce the enterprise production cost by recycling. With
all the results it can be argued that the rotary steam-filtration-adsorption offers a promising route to regenerate and recycle the waste HTF with some certain procedures. Detection results showed that waste oil from manufacturer No. 1, No. 3 and No. 5 can be used in this experiment method for regeneration recycle.

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Reference


