Preparation of Ammonium Metavanadate from A Spent Vanadium Catalyst

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Abstract

• Vanadium compounds are widely used in the chemical industry, e.g. V₂O₅ as catalyst and V₂O₃ as corrosive inhibitor in absorption column of CO₂.
• Up till now, the vanadium sources are titanomagnetcite ores and their concentrates. Researches are conducted to utilize vanadium catalysts that have been disposed in the industrial processes.
• Besides fulfilling the demand for vanadium, the usage of the spent catalysts help to prevent environmental pollution due to the poisonous and dangerous vanadium.
• The objective of this research is to recover vanadium compounds from spent catalyst in form NH₄VO₃. This recovery process consists of two stages, i.e. extraction and crystallization.
• The result shows that the presence of Fe compound has colored and decreased the purity of the crystal. Consequently, Fe compound should be separated by precipitation.

Material

The spent vanadium catalyst were supplied by PT Petromix, East Java, Indonesia. Table 1 shows the chemical analysis of spent catalyst sample.

<table>
<thead>
<tr>
<th>Compound</th>
<th>%w</th>
</tr>
</thead>
<tbody>
<tr>
<td>V₂O₅</td>
<td>5.14</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>1.45</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>1.30</td>
</tr>
<tr>
<td>Na₂O</td>
<td>1.85</td>
</tr>
<tr>
<td>SiO₂</td>
<td>61.29</td>
</tr>
</tbody>
</table>

Extraction by Na₂CO₃ Solution

Spent catalyst (0.002 – 0.0112 gr V₂O₅/m Na₂CO₃)

Extraction 30°C, 60 minutes

Filtration (V₂O₅ - Fe₂O₃)

Residue (solid)

Effect of pH on precipitation of vanadium and iron

The effect of pH on precipitation was studied using different pH in the range 1 – 4 for 2 hours, the results are shown in Figure 3

Preparation of Ammonium Metavanadate

Filtrate from Extraction (V₂O₅ - Fe₂O₃) pH = 10.8

Precipitation 120 minutes

NH₄Cl (11 M)

Catalyst deactivation determining catalyst life.

The deactivated catalyst can be regenerated or reused as a catalyst. This method is the most economic solution. It may involve recovery of V₂O₅ from spent catalyst before disposal at a minimize cost and meeting environmental standards.

The result shows that the presence of Fe compound has colored and decreased the purity of the crystal. Fe compound could be separated by precipitation at pH 12 for two hours and then continued by crystallization of NH₄VO₃ using NH₄Cl 11.215 M for 4 - 5 hours at 60°C.

Introduction

Most chemical processes use catalyst at some stage in production process. The over all catalyst performance is assessed in terms of activity, selectivity and life.

Catalyst which is used in the Contact process, conversion of sulphur dioxide in to sulphur trioxide for production sulphuric acid, contains V₂O₅.

Deactivation of this catalyst may result from poisoning, fouling or coking. The rate of catalyst deactivation determining catalyst life.

The deactivated catalyst can be regenerated or reused as a catalyst. This method is the most economic solution. It may involve recovery of V₂O₅ from spent catalyst before disposal at a minimize cost and meeting environmental standards.

Vanadium compounds from spent catalysts can be recovered in form NH₄VO₃. Compared to V₂O₅, the recovery of NH₄VO₃ is more profitable since the price of vanadate compound in the market is more expensive than V₂O₅. In addition, to produce NH₄VO₃ one can work in neutral condition while the process of obtaining V₂O₅ should be at low pH (1-3). The process in low pH is often avoided since it requires equipment with strict specification.

The recovery process of vanadium from spent catalyst consists of two stages, i.e. extraction and crystallization. The research focused on the crystallization stage and aimed to find an appropriate condition for obtaining NH₄VO₃ crystal with high purity and high recovery percentage.

Analysis Method

• Gravimetric Determining SiO₂ and Al₂O₃ in the spent catalyst
• Titrimetric Determining V₂O₅ and Fe₂O₃ in the spent catalyst, extract and filtrate
• Atomic Absorption Spectrophotometric Determining V, Al, Fe, Na, Si in the spent catalyst, extract and filtrate

Results

The result of extraction by Na₂CO₃ is shown in Table 2.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Concentration, M</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>V₂O₅</td>
<td>0.0300</td>
<td>81.27</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.0550</td>
<td>42.02</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.00007</td>
<td>0.42</td>
</tr>
<tr>
<td>SiO₂</td>
<td>0.00096</td>
<td>0.07</td>
</tr>
</tbody>
</table>

Table 3. Chemical analysis data for AMV product

<table>
<thead>
<tr>
<th>Compound</th>
<th>Composition, %w</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Laboratory</td>
</tr>
<tr>
<td>V₂O₅</td>
<td>75.48</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.01</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>SiO₂</td>
<td>&lt;0.10</td>
</tr>
</tbody>
</table>

Conclusions

• The spent vanadium catalyst could be an alternative resource of vanadium and raw material for ammonium metavanadate
• The optimum conditions for extraction using Na₂CO₃ solution are:
  - 0.006 gram V₂O₅/ml Na₂CO₃ (0.1887 M)
  - time of extraction 60 minutes
• The optimum conditions for ammonium metavanadate preparation are:
  - pH for Fe precipitation is 12
  - temperature crystallization is 60°C

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