# Preparation of Ammonium Metavandate from A Spent Vanadium Catalyst

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## Abstract


- Vanadium compounds are widely used in the chemical industry, e.g. $V_2O_5$ as catalyst and $NH_4VO_3$ as corrosive inhibitor in absorption column of $CO_2$.
- Up till now, the vanadium sources are titomagnetite 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 $NH_4VO_3$. 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.
- $NH_4VO_3$ with 60 %-recovery with respect to the spent catalyst and 76%-purity could be obtained by extraction at room temperature using a solution of $Na_2CO_3$ 1.887 M as a solvent. The time of extraction 60 minutes and the ratio of the $V_2O_5$ weight in the spent catalyst to the solvent volume was 6 gr/l.
- The process was then continued by the precipitation of Fe compound at pH 12 for two hours and the crystallization of $NH_4VO_3$ using $NH_4Cl$ 11.215 M for 4 - 5 hours at 60$^O$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, contents $V_2O_5$.
- 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 $V_2O_5$ from spent catalyst before disposal at a minimize cost and meeting environmental standards.
- Vanadium compounds from spent catalysts can be recovered in form $NH_4VO_3$. Compared to $V_2O_5$, the recovery of $NH_4VO_3$ is more profitable since the price of vanadate compound in the market is more expensive than $V_2O_5$. In addition, to produce $NH_4VO_3$ one can work in neutral condition while the process of obtaining $V_2O_5$ 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 $NH_4VO_3$ crystal with high purity and high recovery percentage.
- 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 $NH_4VO_3$ using $NH_4Cl$ 11.215 M for 4 - 5 hours at 60$^O$C.

## Experimental Works

### Material

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

**Table 1.** Chemical analysis data for spent catalyst

| Compound | %-w |
|---|---|
| $V_2O_5$ | 5.14 |
| $Fe_2O_3$ | 1.45 |
| $Al_2O_3$ | 1.30 |
| $Na_2O$ | 1.85 |
| $SiO_2$ | 61.29 |

### Extraction by $Na_2CO_3$Solution

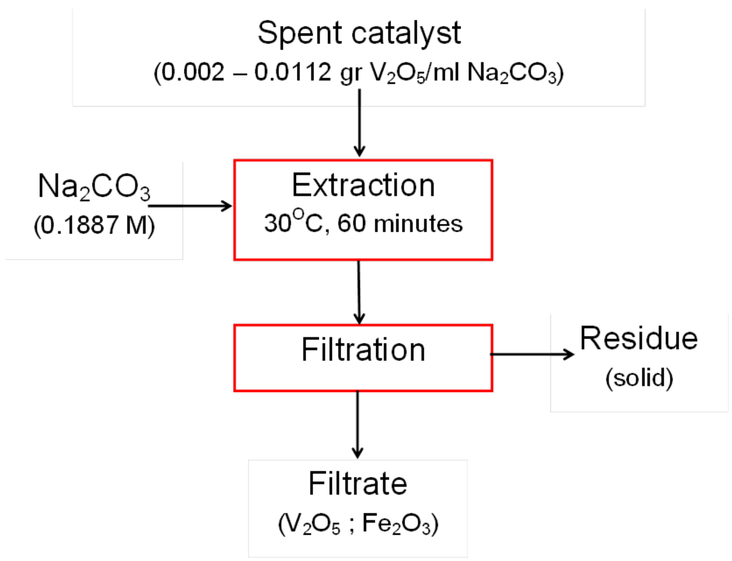


**Figure 1.** Schematic diagram for the extraction of vanadium from Contact Process spent catalyst

### Preparation of Ammonium Metavanadate

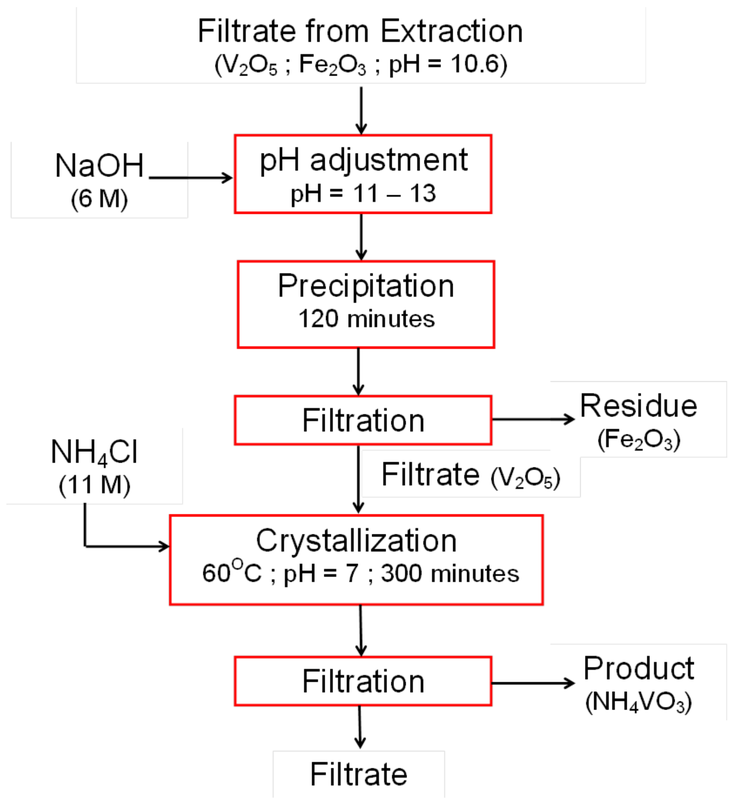


**Figure 2.** Schematic diagram for the preparaation of Ammonium Metavanadate ($NH_4VO_3$)

### Analysis Method

- Gravimetric
  Determining $SiO_2$ and $Al_2O_3$ in the spent catalyst
- Titrimetric
  Determining $V_2O_5$ and $Fe_2O_3$ in the spent catalyst, extract and filtrate
- Atomic Absorption Spectrophotometric
  Determining V, Al, Fe, Na, Si. in the spent catalyst, extract and filtrate

## Results

### Extraction

The result of extraction by $Na_2CO_3$ is shown in Table 2.

**Table 2.** Chemical analysis data for extraction product

| Compound | Concentration, M | Yield (%) |
|---|---|---|
| $V_2O_5$ | 0.03000 | 81.27 |
| $Fe_2O_3$ | 0.00500 | 42.02 |
| $Al_2O_3$ | 0.00007 | 0.42 |
| $SiO_2$ | 0.00096 | 0.07 |

### 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

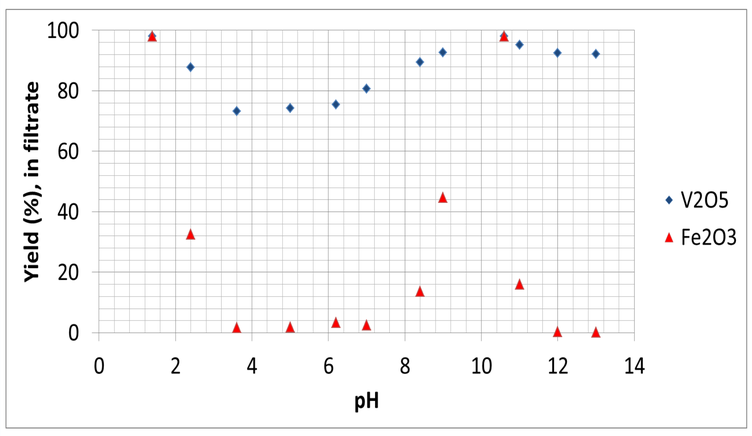


**Figure 3.** Effect of pH on precipitation of vanadium and iron

### Ammonium Metavanadate Preparation

The chemical analysis of precipitation at pH 12 and crystallization at 60$^O$C product indicate that pure ammonium metavanadate was produced. The composisition of this product is similar to ammonium metavanadate commercial, see Table 3.

**Table 3.** Chemical analysis data for AMV product

| Compound | Composition, %-w | |
|---|---|---|
| | Laboratory | Commercial |
| $V_2O_5$ | 75.48 | 76.73 |
| $Fe_2O_3$ | 0.01 | 0.01 |
| $Al_2O_3$ | < 0.01 | 0.01 |
| $SiO_2$ | < 0.10 | 0.10 |

## Conclusion

- The spent vanadium catalyst could be an alternative resource of vanadium and raw material for ammonium metavanadate
- The optimum conditions for extraction using $Na_2CO_3$ solution are :
  - 0,006 gram $V_2O_5$/ml $Na_2CO_3$ (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$^O$C

## Contact :

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