

Novel Eco-friendly Roasting and Leaching Process Development to Recycle Valuable Metals from Spent SCR deNO_x Catalyst

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Research Article

Keywords: Spent SCR Catalyst, Recycle, Roasting, Leaching, Vanadium, Tungsten

Posted Date: July 1st, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-668338/v1>

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Abstract

Spent catalyst, containing vanadium and tungsten oxide in a TiO_2 glass fiber matrix, pose a risk of environmental contamination due to the high toxicity of its metal oxides if leached into the soil when disposed in landfills. Due to the increasing demand of metals and the continuous depletion of primary resources there is an growing necessity for recycling and reprocessing of spent catalysts and other secondary metal sources for environmental and economical reasons.

Study of spent SCR catalyst soda roasting process with dissolved NaOH compared with the usual NaOH dry roasting and its influence in the subsequent water leaching. After optimization, the ideal parameters are roasting with 40% NaOH solution for 2h at 973K and DI water leaching for 30minutes, at 298K with a pulp density of 30%. The research results show an important reduction of the roasting temperature and leaching time during the processing of spent SCR catalyst with the objective to recover vanadium and tungsten. Silicon compounds are one of the main impurities leached alongside the valuable metals and in this work, the silicon compounds leached are reduced significantly avoiding the de-silication post-processing of the leach liquor. The main advantage of the proposed process is the increase of the leaching efficiency of vanadium and tungsten in a shorter time regardless of the leaching temperature.

Introduction

Since the 2000s (new millennium), the demand for selective catalytic reduction (SCR) catalysts for the minimization of the NO_x discharged from a variety of sources (stationary and mobile) has been increasing worldwide as concerns about environmental pollution caused by nitrogen oxides have increased¹⁻³. The amount of waste catalysts generated by increasing the amount of SCR catalysts is also increasing^{4,5}. Until now, waste catalysts that have been deactivated by poisoning have been reused through the regeneration process, but when the activity is reduced, most of them are disposed in landfills with designated waste⁶⁻⁸. However, the composition of the spent catalyst can be analyzed and it can face another environmental threat when it is disposed or buried due to the accumulation of V_2O_5 , a highly toxic compound, and heavy metals. In addition, the catalyst after use cannot be directly reprocessed as a new catalyst carrier because it has high content of As, Na, Ca, Fe and K and is very vulnerable to chemical toxicity⁹⁻¹¹. SCR catalysts consisting of $\text{V}_2\text{O}_5\text{-WO}_3/\text{TiO}_2$ generally have very high Ti, W and V content, so it can be an economic and environmental feasible option to recover and reuse them. Ti, W, and V remanufactured after recovery can be used as raw materials for new catalysts or as raw materials in other industries^{4,12,13}. Accordingly, technology development studies are actively being conducted to recover and remanufacture raw materials from waste catalysts¹⁴. Wet smelting methods for the recycle of titanium, tungsten and vanadium from spent SCR denitrification catalysts are widely known, which are prepared by leachate such as soda-roasting¹⁵, pressurized leaching¹⁶, and acid leaching, and then purified rare metal ions in the solution¹⁷.

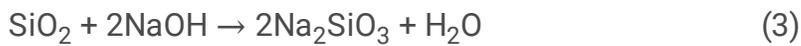
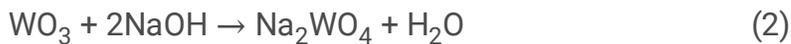
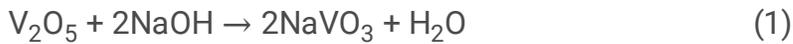
Wu et al. (2016) studied the reprocessing of tungsten from SCR spent catalyst (honeycomb type) by alkali leaching-ion exchange method. The waste catalyst samples of 74 μm particle size were leached at a high liquid ratio of 3% and a reaction temperature of 70°C for 30 minutes, respectively, and 91 wt% and 87 wt% of W and V were leached, respectively. The leachate was adsorbed using a strong base anion exchange resin (Amberlite IRA900), and the divalent WO_4^- was selectively separated under high pH conditions¹⁸. Choi et al. (2019, 2018a) performed soda-roasting experiments with Na_2CO_3 as a scavenging agent from SCR spent catalysts. The amount of Na_2CO_3 was 10 equivalent and the particle size of less than 106 μm was distributed at 1070K for 120 minutes. In this study, it was found that the increase of the amount of tungsten leached was significantly related to the inhibition of CaWO_4 production due to the increase of Na_2CO_3 addition along with the rate of TiO_2 anatase changing phases to rutile. In contrast, the amount of vanadium leached was not influenced by soda blasting, and the results of the experiment showed an approximate constant rate of about 40%. This was attributed to the production of calcium vanadium acid when reacting with CaO from raw material, and the leaching ability of V was obtained analyzing the amount of calcium in the raw material^{2,10,15}. Wu et al. (2018) examined the selective leaching and reaction mechanisms of V and Fe using oxalic acid. The experiment was performed for 180 minutes under the condition of a reaction temperature of 90°C, the high liquid ratio of 20 mL/g, and the particle size of 75 μm using 1.0 mol/L concentration of oxalic acid, and 84% of V and 96% of Fe were leached. The soluble cations of VO_2^+ and Fe^{3+} were reduced through the dissolution and complexation process through leaching reaction. V and Fe were found to be highly leaching when they were present in certain forms of VOC_2O_4 and $\text{Fe}(\text{C}_2\text{O}_4)_2$ at 0.33 pH, which showed that the oxidation reduction reaction resulted in the destruction of the dissolution and complexing equilibrium for VO^{2+} , VO^+ and Fe^{3+} . In case the of tungsten and titanium, only the dissolution and complexation reaction occurred and the leaching efficiency was hindered by solubility¹⁹.

However, in previous studies, if a large amount of SiO_2 is leached together with vanadium and tungsten, there is a competitive leaching process where vanadium and tungsten leaching could be inhibited by the presence of silicon. In addition, after leaching for separation of the title metals by ion-exchange or solvent extraction, silicon must be removed adding an extra step to the recovery and purification process^{15,20-22}.

In this study, we performed a hydrometallurgical process through soda roasting and water leaching for the recovery of vanadium (V) and tungsten (W) from spent SCR de NO_x catalysts. The leaching efficiency of vanadium, tungsten and silicon was compared based on the premise that the phase of the roasting agent, either NaOH solid or NaOH solution, affects the leaching conditions of the title metals. The optimum leaching condition is derived based on the roasting agent amount, temperature and time. For water leaching the parameters to consider are solid liquid ratio, the reaction temperature, and reaction time.

Results And Discussion

In the soda roasting process, vanadium and tungsten are converted into the water-soluble compounds NaVO_3 and Na_2WO_4 as shown in reaction 1 and 2. However, in the spent SCR catalysts, in addition to vanadium and tungsten, acting as catalysts, there is SiO_2 contained in the glass fiber matrix which improves the mechanical properties of the catalyst, therefore Na_2SiO_3 is generated (reaction 3) during the roasting process. Na_2SiO_3 is leached alongside V and W inhibiting the complete leaching of the title metals. As a result, the amount of vanadium and tungsten leached decreases due to the presence of silicon soluble compounds. Therefore, an experiment was performed to reduce the leaching rate of SiO_2 while reaching the optimum leaching conditions for vanadium and tungsten.



NaOH phase and temperature effect.

In the soda roasting process, the reaction temperature is a very important factor in the conversion of V_2O_5 and WO_3 to NaVO_3 and Na_2WO_4 from the feedstock. An experiment was conducted to determine the effect of the phase and reaction temperature of NaOH used as a roasting agent in the amount of vanadium, tungsten and silicon present in the leaching solution. 15g of NaOH were added as a solid to the SCR spent catalyst sample and roasted at 773 ~ 1173 K for 2 hours while 15g NaOH were dissolved in a sufficient amount of water and subjected to the same roasting temperatures. After roasting, the residue leached in 100 mL of distilled water at 298 K for 3 hours. As shown in Fig. 1(a), the leaching efficiency of vanadium and tungsten augmented as the roasting temperature increased. In the case of dissolved NaOH, the leaching rate was very low at 773 K, but it increased significantly at 873 K, and it was found to reach the maximum at 973 K. On the other hand, when the roasting agent is solid NaOH, the reaction starts after melting resulting in a lower leaching efficiency of the title metals. Moreover, dissolved NaOH has superior mass transfer activity with vanadium and tungsten in the waste catalyst and the ionized sodium ions react more evenly with the waste catalyst particles than NaOH in the molten state. Fig 1 (a) shows that the leaching ability of vanadium and tungsten is maximized when the roasting agent is NaOH solution, while Fig 1 (b) shows that at the same roasting temperature (973K) the concentration of silicon leached in the solution is almost half in the case of NaOH solution used as a roasting agent.

Effect of sodium hydroxide concentration.

An experiment was conducted by adding the same mass of SCR spent catalyst with an identical volume of NaOH solution at different concentrations ranging from 10 to 50% to compare the leaching efficiency of vanadium and tungsten. The mixture of NaOH aqueous solution and feedstock was roasted in a

muffle furnace for 2 hours at 973K, and after roasting; the sample was added to distilled water and leached at 278 K for 3 hours. The amount of vanadium and tungsten leached according to each experimental condition is shown in Fig. 2 (a). As the concentration of NaOH augmented, the leaching efficiency of vanadium and tungsten was greater. When the concentration was more than 10%, the leaching rate increased significantly, and when the concentration was 40%, the leaching efficiency of tungsten reached a maximum, while vanadium leaching increase was insignificant. Therefore 40% NaOH is considered as the optimum concentration.

Effects of roasting time.

To compare the leaching efficacy of vanadium and tungsten according to the roasting time, roasting was performed for 30 to 180 minutes, followed by leaching. As shown in Fig. 2 (b), leaching efficiency of vanadium increases as the roasting time increases, and the tungsten leaching reaches a maximum after 120 minutes. Due to the low concentration of vanadium in the original sample, 120min is established as the ideal roasting time.

Effect of pulp density for leaching.

Various experiments were used to examine how the pulp density affects the leaching efficiency of V and W. Leaching experiments were performed at 298 K and 300 rpm for a sufficient time to have complete leaching (120minutes) at 10-50% pulp density. As shown in Fig. 6, the pulp density does not have a great influence in the leaching efficiency; still, the highest leaching efficiency was at 30% pulp density. While, above 40%, the leaching efficiency decreases. It is expected that if the amount of spent catalyst used is higher, the concentration of vanadium and tungsten leached will be higher too, however, as noticed in Fig. 3 (a) the leaching efficiency does not improve with the increase of the pulp density. Thus, for an ideal mass transfer 30% pulp density was set as the optimum condition.

Effect of leaching time.

The effect of the leaching time from 10 to 180 minutes during the leaching process in the leaching efficiency of vanadium and tungsten was explored. After soda roasting at 973 K for 120 minutes, 100 mL of distilled water was used for leaching from 10 to 180 minutes at 298 K, 300 rpm, and 30% pulp density. As shown in Fig.3 (b), the leaching time does not contribute significantly to the leaching efficiency for vanadium and tungsten. Several researchers have studied the ideal conditions for roasting and leaching of spent SCR catalyst, however time leaching time has always been long (at least 1h) to maximize the leaching of vanadium and tungsten^{19,20,23,24}. For instance, Moon et al obtained an almost complete leaching of vanadium and tungsten in a 1h interval²⁵. Wu et al did an alkaline leaching without a preprocessing (soda roasting) of the catalyst which possesses the disadvantage of a large consumption of alkali in the process¹⁹. The leaching system studied in this investigation reaches a maximum in 30

min, which makes not only savings in the economic part of the system but also in the efficiency point of view due to the reduced leaching time. It is deduced that vanadium and tungsten were converted into a form that facilitated leaching during the soda roasting process using NaOH aqueous solution.

Effect of temperature for leaching.

In the leaching process, the reaction temperature affects the mass transfer activity of the water-soluble target metal and acts as an important factor to determine the amount of metals leached. The effect of leaching temperature on leach efficacy of vanadium and tungsten was studied in a range between 298 to 343 K. As shown in Fig. 3 (c), the leaching efficiency for both title elements does not show a significant response to the temperature increase in the leaching process, which lead to the conclusion that to save resources 298K will be used as the optimal leaching time. Moreover, it is believed that as the reaction temperature increases, the activities of substances other than vanadium and tungsten in the spent catalyst increase, therefore a lower temperature (298K) is preferred to avoid an increase in the leaching of silicon or any other impurity.

Morphology of the samples at different metal recovery stages.

SEM-EDS images were taken for the raw spent SCR catalyst, after roasting and after leaching to compare the morphology of the residues at different stages. Figure 4 shows the SEM images at different magnifications for the original spent SCR catalyst, the spent SCR catalyst roasted with dissolved NaOH and the residue after leaching. Fig 4 (a1), (b1), (c1) show the spent catalyst sample before roasting, after roasting and after leaching at low magnification, respectively. It can be observed that in the case of the raw spent catalyst (a1) there is a clear presence of fiber glass rods (silica, alumina and calcium oxide) which are the main concern for this research due to the minimization of silica leaching as a primary research goal. As observed in the posterior EDS figures it is clear that the granules surrounding the glass fiber rods are composed mainly of titanium oxide (anatase form). In Fig 4 (b1) and (c1) the rods are not clear in shape at low magnification due to the crushing with mortar and pestle done to the roasted residue.

When the magnification is increased 1000 times, it can be observed that in Fig 4 (a2) and Fig 4 (b2) there is some agglomeration on the rod type structures while in Fig 4 (c2) the rod structures show a smooth texture. In the largest magnification Fig 4 (a3), (b3) and (c3), it can be observed in the original spent catalyst the absence of pores, which is characteristic of a deactivated catalyst. Moreover, in Fig 4 (b3) there is agglomeration over the rod-type structure, which can be related to the formation of sodium compounds on the fiberglass matrix. Finally in Fig 4 (c3), a smooth texture is observable characteristic of fiber glass surface, which leads to the deduction that vanadium and tungsten sodium compounds were

leached from the surface leaving the fiber glass (composed of calcium, aluminum and silicon) in its original shape.

In Fig 5 corresponding to the EDS analysis of the raw spent SCR catalyst it can be appreciated that there is a clear silicon and calcium rod structure in the middle covered mainly in tungsten. Moreover, vanadium and titanium are spread along the sample, the presence of titanium around the rod type structures and spread is in accordance with the main component of SCR catalyst that is TiO_2 (anatase phase)

Fig 6 shows the catalyst EDS analysis after roasting, an agglomeration of sodium particles can be observed covering the fiber glass rod which indicate the formation of sodium compounds such as sodium vanadate and tungstate. However, due to the large presence of sodium all over the rod-like structure, it can be deduced that Na_2SiO_3 might have been formed. Moreover, the percentage of sodium in the sample has increased in accordance to the reaction with the metals present.

It is observable in the EDS patterns in Fig 7 that after leaching the density of sodium particles present in the sample decreases, while the percentage of vanadium almost keeps constant (due to the small initial presence of this element) but tungsten decreases significantly. Moreover, the percentage of silicon compounds varies, which is in accordance to the results obtained in the roasting and leaching parameters analyzed previously. In addition, the morphology of the sample keeps the rod-type structure with small agglomeration of particles on top indicative of vanadium and tungsten not leached during the process.

Experimental

Feedstock.

The raw spent SCR catalyst was obtained from the thermoelectric power plant in Samcheonpo, South Korea. The samples were grinded to a size of less than $100\ \mu\text{m}$, in addition to removing and crushing the dust and contaminants on the surface before experiments. The crushed waste catalyst samples were desiccated at 100°C for a day. The composition of the feedstock used in the experiment is shown in Table 1, and the XRD diagram is shown in Fig. 8.

Table 1

Elemental composition of spent SCR catalyst.

| Component | TiO_2 | SiO_2 | WO_3 | Al_2O_3 | CaO | V_2O_5 | FeO | MgO | MoO_3 | H_2O |
|-----------|----------------|----------------|---------------|-------------------------|------|------------------------|------|------|----------------|----------------------|
| Wt.% | 70.9 | 9.80 | 7.73 | 5.57 | 2.50 | 1.23 | 0.77 | 0.55 | 0.10 | 0.85 |

Soda roasting and leaching:

Sodium hydroxide (NaOH, supplied by Junsei Chemical, 99% purity, Japan) was used for the soda roasting of the spent SCR deNO_x catalyst. NaOH reacts with insoluble V₂O₅ and WO₃ to obtain soluble compounds (NaVO₃, Na₂WO₄) by soda roasting from spent SCR deNO_x catalysts. In order to compare the efficiency according to the state (solid or liquid) of NaOH used as a roasting agent, solid NaOH was grinded and mixed with the spent SCR catalyst and placed in a alumina crucible. On the other hand, dissolved NaOH was mixed with the spent SCR catalyst and stabilized for 10 minutes to wet the solid totally in an alumina crucible in preparation for the roasting process. The amount of NaOH added was 10 to 50% by weight, depending on the weight of the feedstock, and roasted in a muffle furnace without any gas or air inlet for 2 hours while varying the temperature conditions. The roasting experiments were performed at temperatures ranging 773 to 1173 K and time variation from 30 to 180 minutes to understand the effect of the reaction temperature and reaction time. After soda roasting, the cooled sample was placed in a Teflon reactor and leaching experiments were performed on a hot plate modifying different conditions such as pulp density, temperature and reaction time. The overall environmentally friendly process for spent SCR catalyst is depicted in Figure 9.

Analysis.

To obtain the concentration of the title elements (V, W, Si) in the leach liquor an inductively coupled plasma optical emission spectrometer (ICP-OES, Optima 8500, USA) was used. The composition of the raw sample and residues of spent SCR catalyst was analyzed by X-ray diffractometer (XRD, Rigaku, Japan). The scanning electron microscope and energy Dispersive Spectrometer (SEM-EDS, JSM-6380LA, Japan) was used to analyze the surface and composition of samples.

Conclusion

Due to the environmental risk that spent SCR catalyst pose when discarded and the presence of valuable metals such as vanadium and tungsten, there is a big necessity for recycling of these secondary resources. During the present research the roasting process of spent SCR catalyst was optimized comparing NaOH dry roasting and aqueous roasting using NaOH solution as a roasting agent. The main goal of the investigation is to maximize the leaching efficiency of both metals while minimizing the silicon compounds leached into the pregnant solution. The optimum conditions for roasting are 40% concentration NaOH solution for 2h at 973K. With a subsequent DI water leaching for 30minutes, at 298K with a pulp density of 30%. At the optimum conditions there was a leaching efficiency of $95.4 \pm 3\%$ of tungsten while the vanadium leaching efficiency ranged in $80.2 \pm 3\%$. Research results showed that using NaOH solution as the roasting agent reduced the posterior leaching of silicon compounds and it was observed that at the optimal roasting conditions the amount of silica leached was 1.6 times smaller than when using dry NaOH as a roasting agent.

Declarations

Acknowledgements

This study was supported by the R&D Center for Valuable Recycling (Global-Top R&D Program) of the Ministry of Environment (Project Number: 2019002230001), Korea.

Author contributions

Dr. Jong Hyuk Jeon did all the experiments and wrote the manuscript. Ms. Ana Belen Cueva Sola did the formal analysis of the SEM and SEM-EDS data, Prof. Jin-Young Lee and Prof. Rajesh Kumar Jyothi supervised the experiments, did the formal analysis, and edited the manuscript.

Additional Information

Competing Interests: The authors declare no competing interests.

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Figures

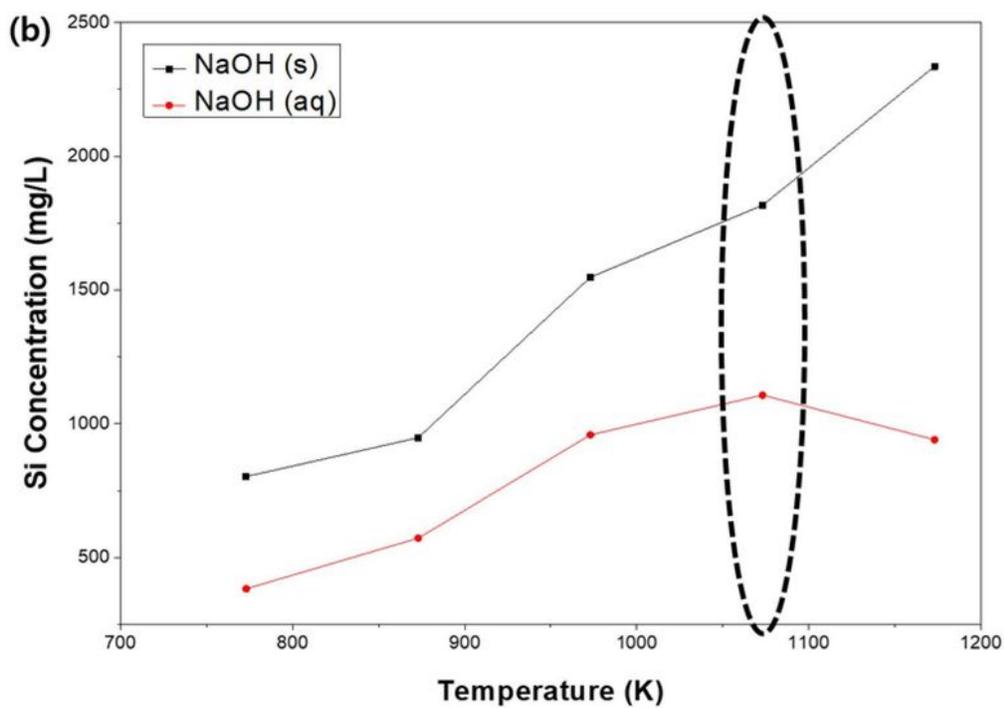
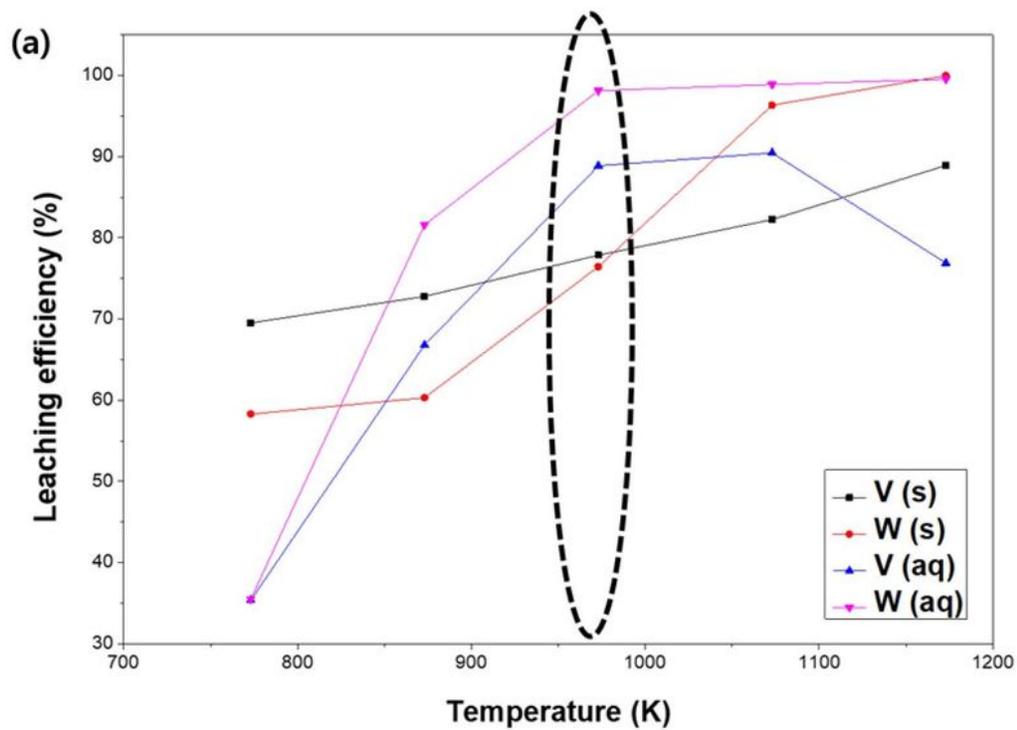


Figure 1

Effects of temperature at the time of roasting process (s = solid, aq = aqueous) in the (a) leaching of vanadium and tungsten and (b) in the concentration of Si leached

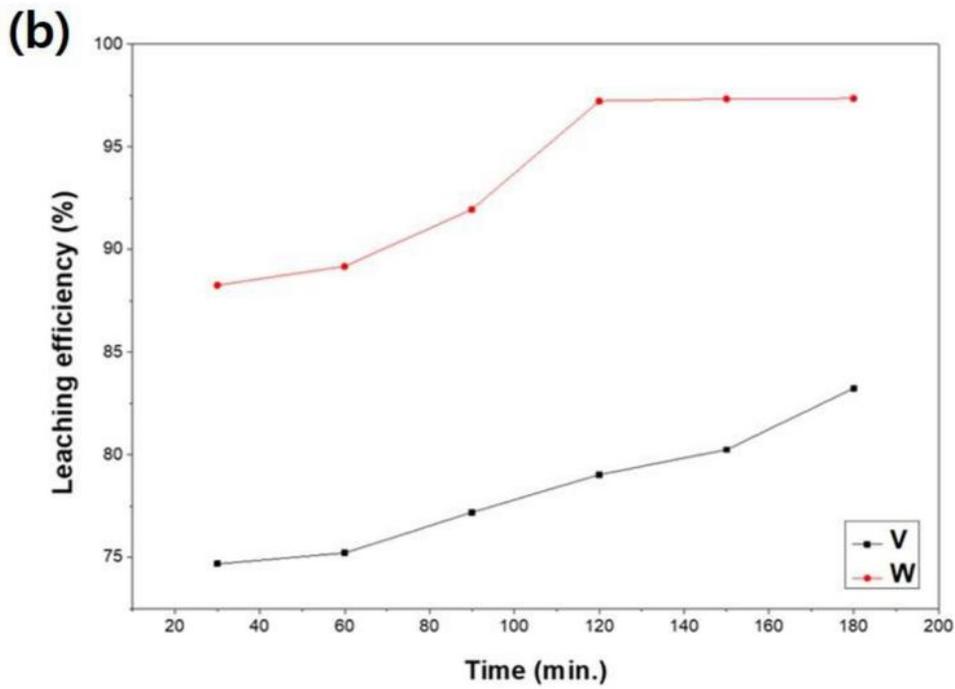
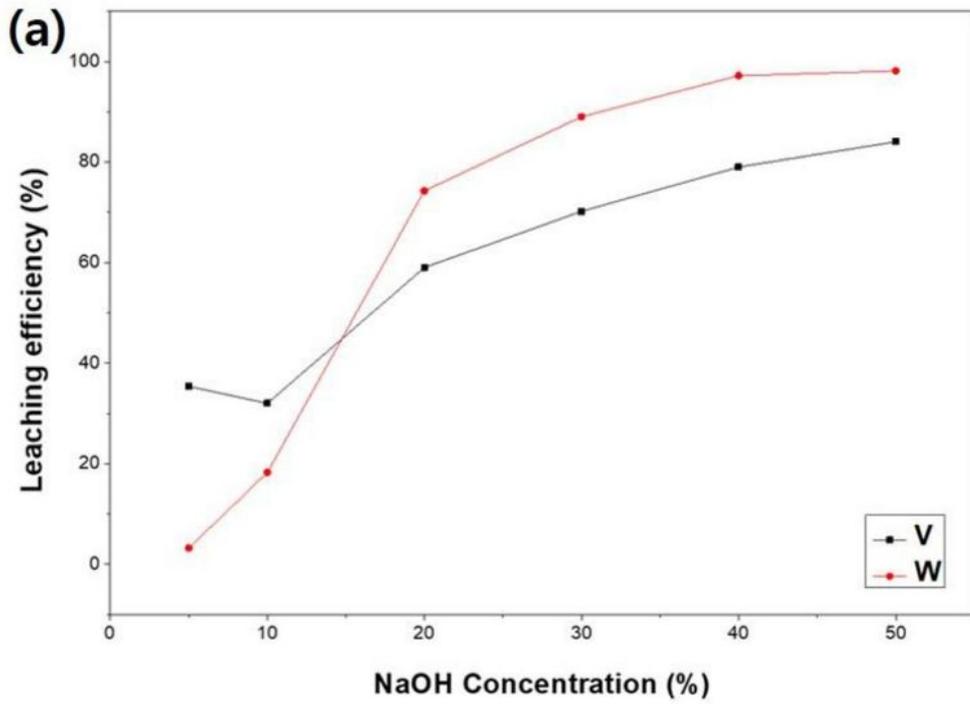


Figure 2

Effect of sodium hydroxide concentration and time for roasting process

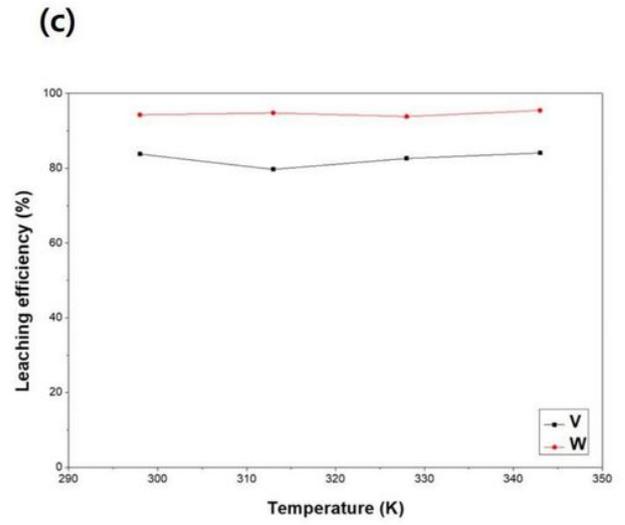
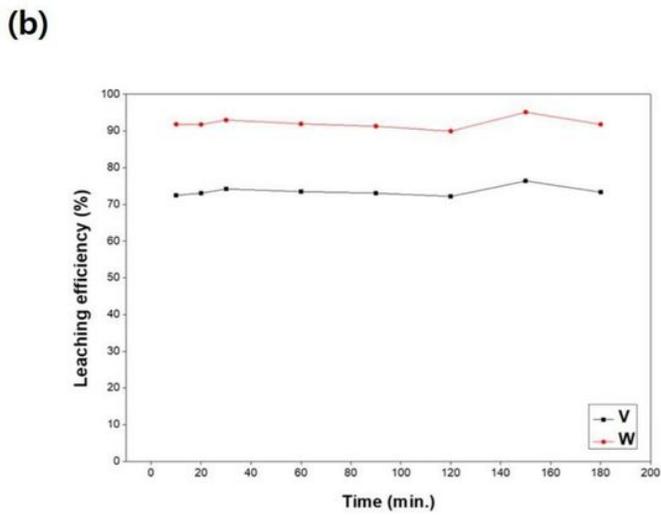
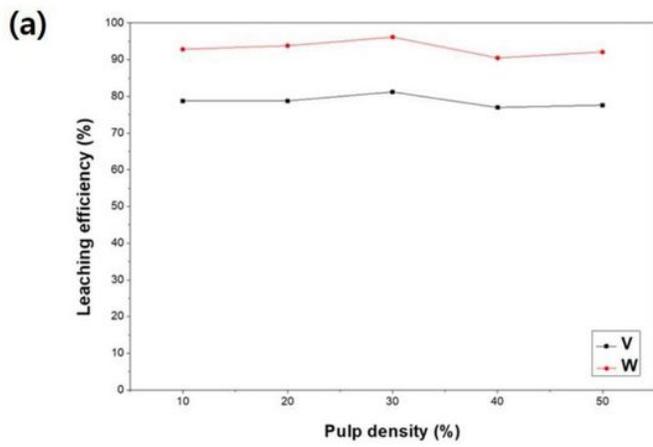


Figure 3

Effect of pulp density, time, temperature for leaching of the spent SCR catalyst

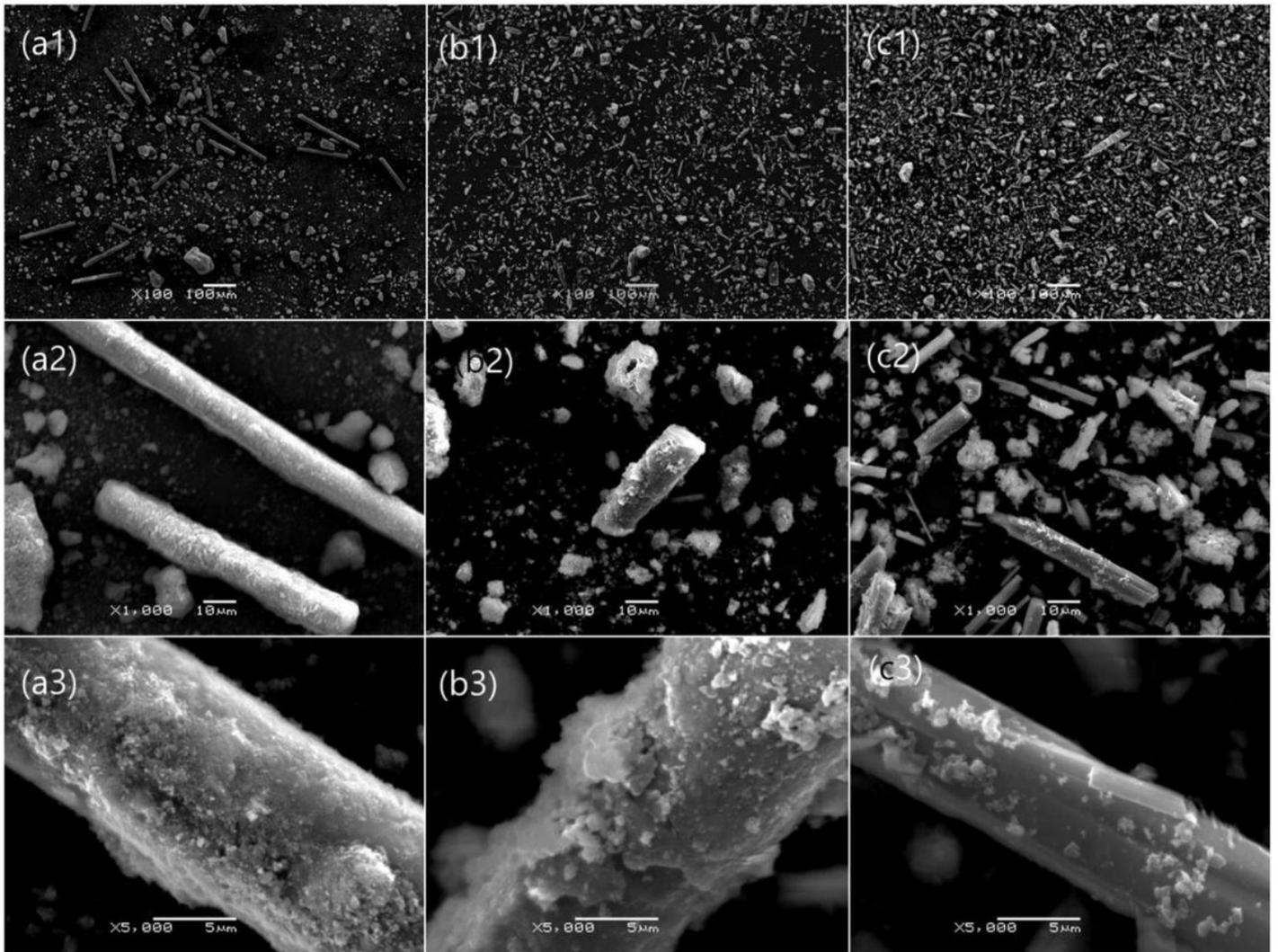


Figure 4

SEM images of (a) raw spent SCR catalyst, (b) soda roasted spent SCR catalyst and (c) residue obtained when water leaching was conducted at 298 K

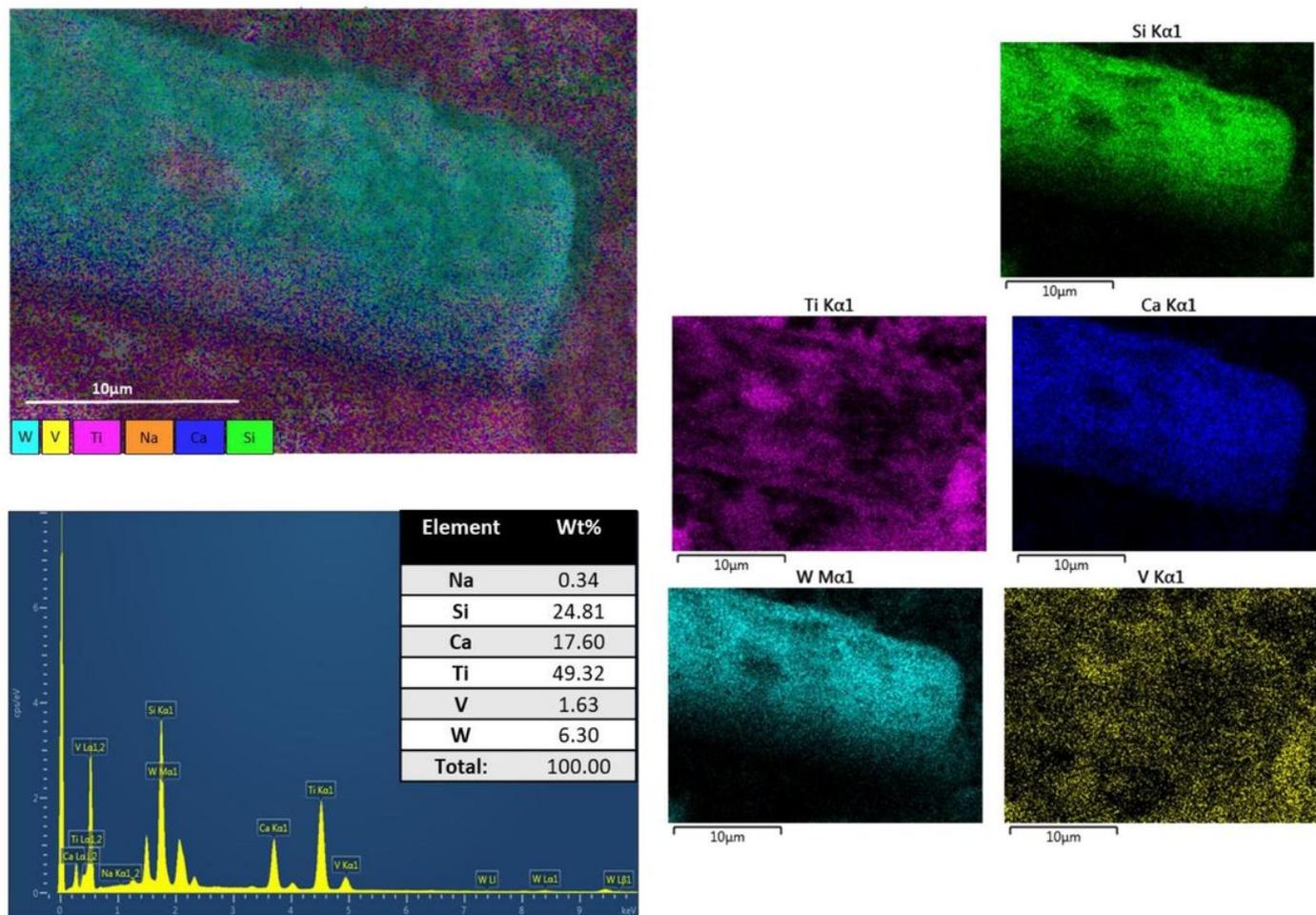


Figure 5

SEM-EDS images and element composition of raw spent SCR catalyst

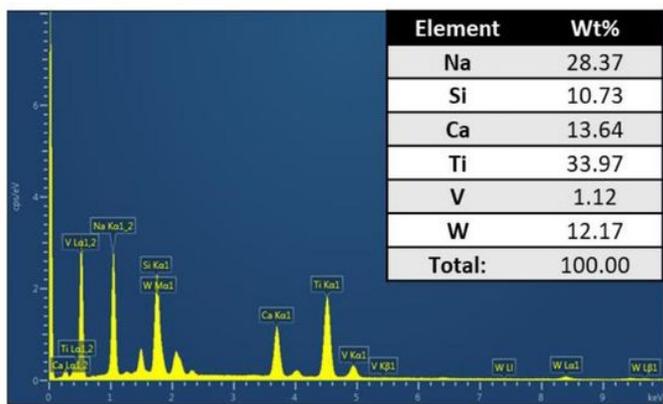
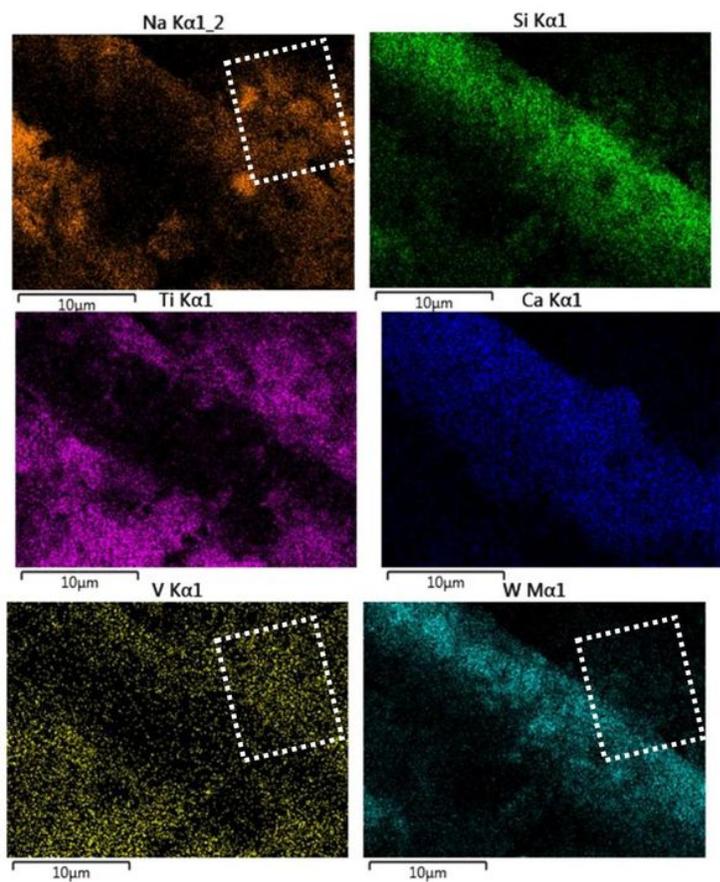
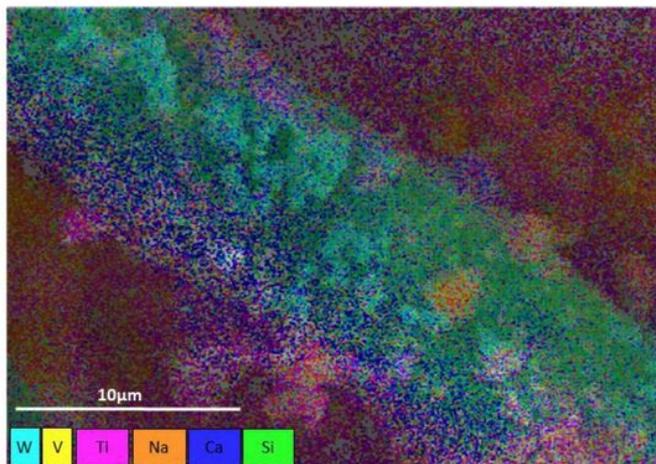


Figure 6

SEM-EDS images and element composition of roasted spent SCR catalyst

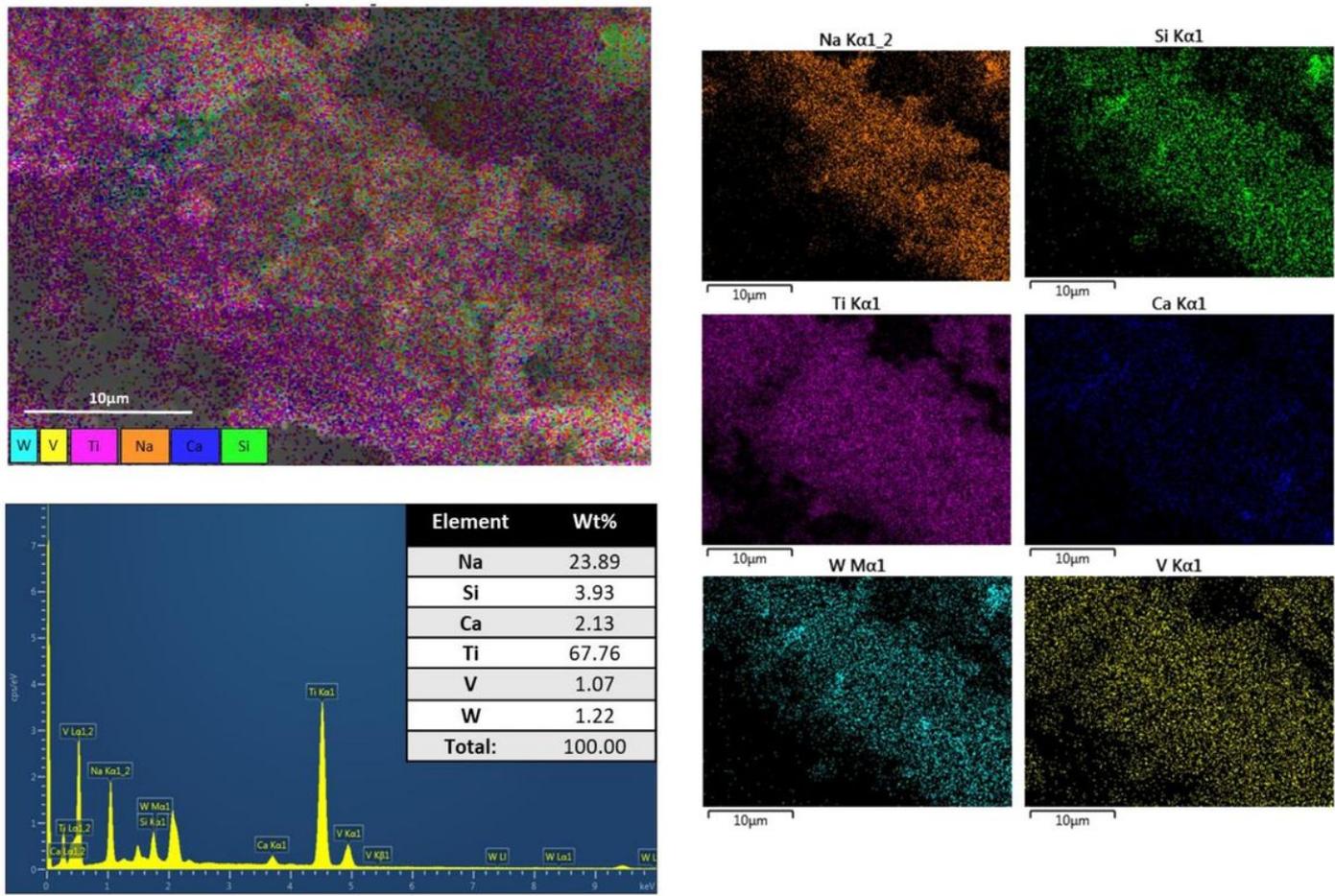


Figure 7

SEM-EDS images and element composition of leached catalyst

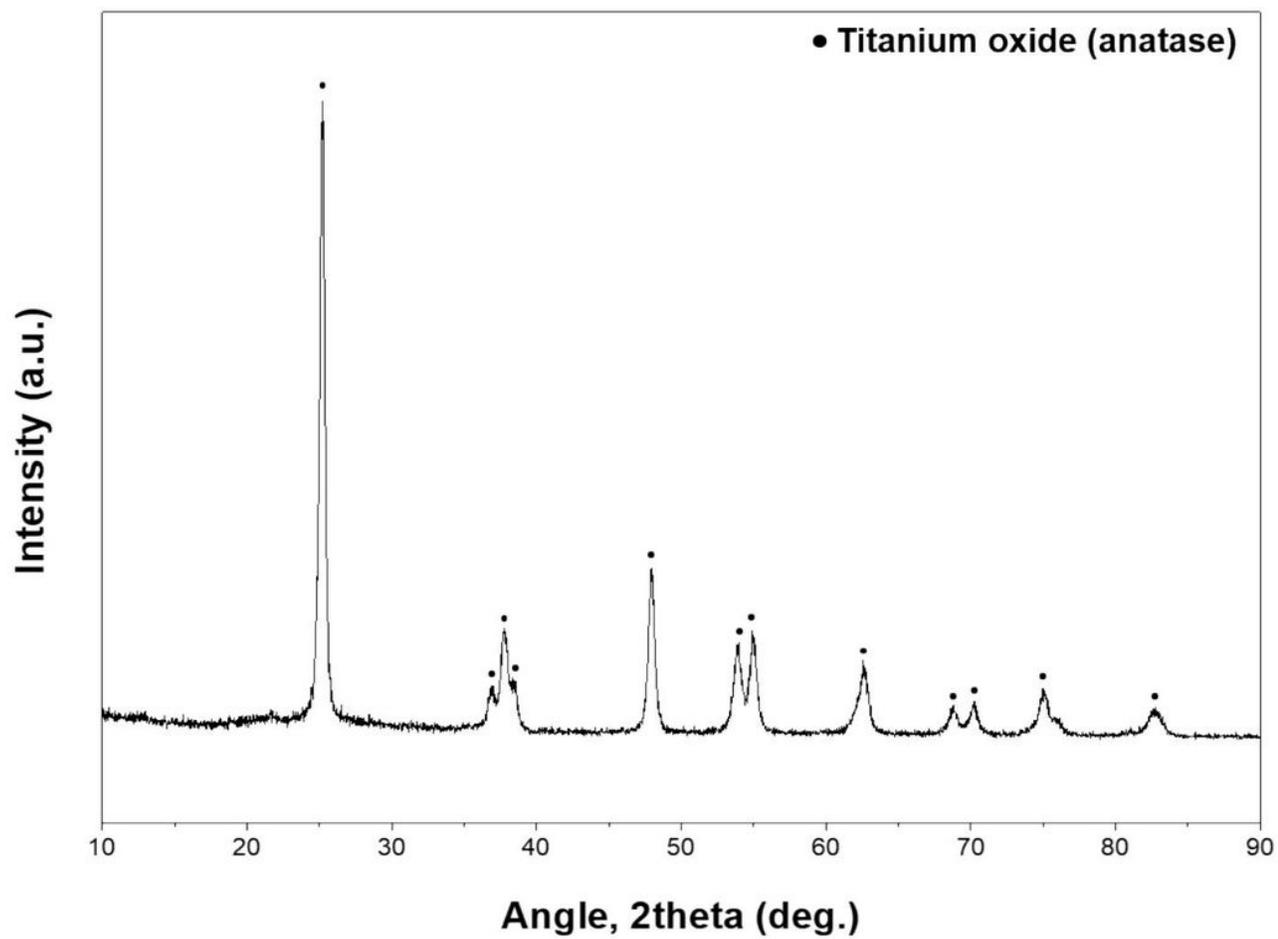


Figure 8

XRD pattern of spent SCR catalyst

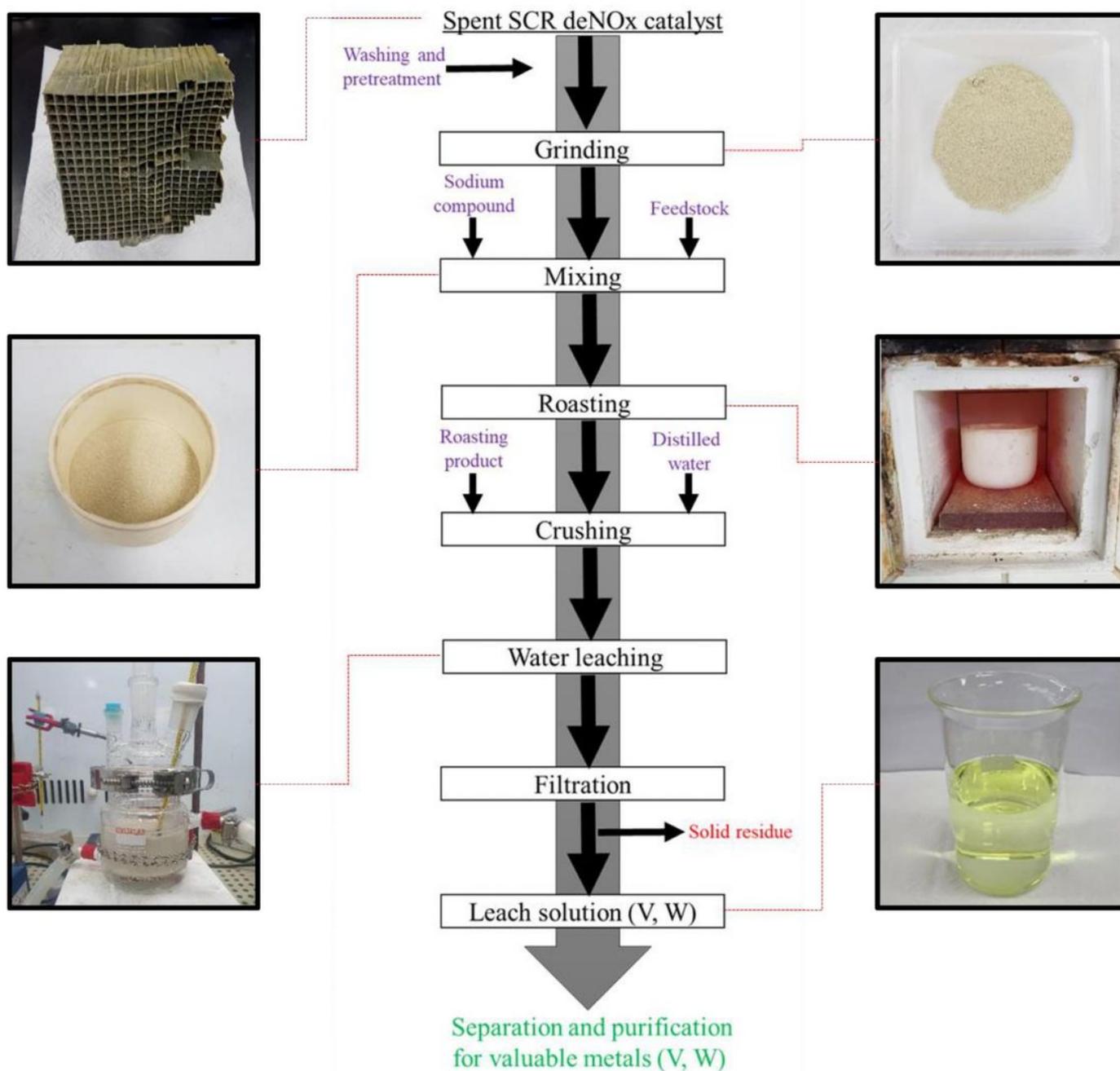


Figure 9

Eco- friendly hydrometallurgical process flowsheet for spent SCR DeNOx catalyst leaching