Simultaneous leaching of Pt, Pd and Rh from automotive catalytic converters in chloride-containing solutions

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Abstract. Dissolution of platinum group metals (PGM; herein Pt, Pd and Rh) in different chloride-based leaching systems from spent auto catalysts was performed. Response surface methodology and a five-level-five-factor central composite design were used to evaluate the effects of 1) temperature, 2) liquid-to-solid ratio, 3) stirring speed, 4) acid concentration and 5) particle size on extraction yield of PGM by aqua regia. Analysis of variance was used to determine the optimum conditions and most significant factors affecting the overall metal extraction. In the optimum conditions, leaching of Pt, Pd and Rh was 91.58%, 93.49% and 60.15%, respectively. The effect of different oxidizing agents on the PGM dissolution in chloride medium was studied comparatively in the following leaching systems: a) aqua regia/sulfuric acid mixture, b) hydrogen peroxide in sulfuric acid (piranha solution), c) sodium hypochlorite and d) copper(II). Dissolution of Rh is increased in both aqua regia and hydrogen peroxide/hydrochloric acid solutions by adding sulfuric acid.

1. Introduction
The primary use of PGM is in the automobile industry. In the first half of 2016, the amount of PGM used in automobile catalysts as a percentage of total world demand for Pt, Pd and Rh was 40.3%, 78.7%, and 81.5%, respectively [1]. In a catalytic converter, PGM nanoparticles are evenly coated onto a ceramic honeycomb substrate [2,3]. Sintering, thermal deactivation and vehicle vibration can lead to deterioration of catalyst [2], because PGM particles gradually grow in size mainly by Ostwald ripening [4] and can be oxidized for instance in the case of Rh to form Rh₂O₃ [5]. Hence, replacement of catalytic converter is needed. As a result, automotive industries produce annually considerable amounts of spent catalysts containing PGM. Owing to their extensive use in catalysis, electronic devices and jewellery, among others, PGM demand is increasing. In addition to high production costs from naturally occurring supplies, low production of these metals due to their low abundance and complexity of PGM mineralogy in the Earth's crust [6], has made precious metals extraction from spent catalysts a viable and cost effective operation [7].

Aqua regia has been frequently used to dissolve PGM mainly due to rapid leach rates and high metal recoveries [8]. However, process needs further improvement due to consumption of both acid and complexing agent in side reactions, such as dissolution of alumina and other oxides. A lack of knowledge still exists in deeply understand the oxidative dissolution of Pt, Pd and Rh in solutions containing chloride ions [9]. The difficulty of using a phenomenological model may be surmounted by the use of a statistical model, to predict the best conditions for the leaching system within the typical range of representative process variables. Therefore, the aim of experimental design here used was to
maximize the recovery of the PGM all at once, taking into account the ranges of five variables and interactions among these. Optimum results obtained in the leaching by aqua regia are compared to the results achieved by using HCl and other oxidizing agents.

2. Experimental

2.1. Preparation of sample
About 800g of PGM-containing honeycomb type support of one spent automobile catalysts was used. The ceramic support was crushed, dried and sieved. Chemical analysis of particles sample was determined by inductively coupled plasma atomic emission spectroscopy (Varian Liberty RL ICP-AES). X-ray fluorescence spectrometer (PW2404, Philips) was used for the analysis of support.

2.2. Leaching experiments
Solutions were prepared in ultra-pure water (resistivity 18.2 MΩ). Analytical grade chemicals were used without further purification. Aqua regia was freshly prepared by mixing concentrated hydrochloric acid (37%) and concentrated nitric acid (65%) at a volume ratio of 3 HC l:1 HNO₃. Hydrogen peroxide, solution 30% w/w (110vol), reagent grade was used. The batch reactor used was a 0.5L glass container equipped with a thermometer, a mechanical turbine-type stirrer and a flux condenser. The condenser was used to recover and send back to reactor the vapour. Any escaped gases (such as Cl₂, NOCl or NOx) at the exit of the condenser were scrubbed into a cold water container. Temperature was kept constant using a water bath. All analyses of PGM were done using an atomic absorption spectrophotometer (Varian-AA240).

2.3. Experimental design
A five-level-five-factor central composite rotatable design was used, requiring 32 experiments in the factorial portion, 10 in the axial points, and 3 center experiments for 45 experiments. Response surface methodology (RSM) was used for statistical optimization of leaching process for the extraction of PGM, which is influenced by number of operating parameters with a minimum number of experiments as well as to analyse the interaction between the parameters. Statistical design-expert software DX7 was used to analyse the significance of the experimental results. The experimental variables and their corresponding levels are listed in Table 1. Five different parameters were varied: HCl concentration from 6M to 13M, temperature from 25°C to 90°C, sample size from -53 to -106µm, liquid-to-solid ratio from 2 to 10 mLg⁻¹ and stirring speed from 100 to 400rpm.

| Table 1. Experimental range and levels of the independent variables. |
|---------------------------------------------------------------|
| | Low axial (-α=2) | Low factorial (-1) | Centre (0) | High factorial (+1) | High axial (+α=+2) |
| HCl concentration (M) | 6 | 7.4 | 9.5 | 11.5 | 13 |
| Temperature (°C) | 25 | 38.6 | 57 | 76.3 | 90 |
| Liquid/solid (mLg⁻¹) | 2 | 3.6 | 6 | 8.3 | 10 |
| Particle size (µm) | 53 | 64.1 | 79.5 | 94.8 | 106 |
| Stirring speed (rpm) | 100 | 163 | 250 | 337 | 400 |

3. Results and discussion

3.1. Chemical composition of spent auto catalysts
The data in Table 2 reflect the typical composition of a honeycomb-type automobile catalysts [3], which is made of cordierite (2MgO·2Al₂O₃·5SiO₂), coated with alumina (Al₂O₃) and a combination of PGM (Pt, Pd, Rh), rare earth oxides (La₂O₃, CeO₂) and other oxides.
Table 2. Major and trace elements concentrations in spent catalytic converter.

| Oxide | MgO | Al₂O₃ | SiO₂ | CaO | Cr₂O₃ | Fe₂O₃ | NiO | ZnO | ZrO₂ | BaO | La₂O₃ | CeO₂ |
|-------|-----|-------|------|-----|-------|-------|-----|-----|------|-----|-------|------|
| wt%   | 9.8 | 38.15 | 34.28| 1.16| 4.56  | 3.90  | 1.09| 1.85| 0.73 | 0.84| 2.73  |
| Element | Pt | Pd | Rh |
| ppm   | 914 | 876 | 114 |

a Results obtained by X-ray fluorescence (XRF) analysis.
b Results for Pt, Pd and Rh after digestion were obtained by ICP-AES.

3.2. Optimization of aqua regia leaching process

Leaching experiments of sample were performed with aqua regia during 4h. The variables investigated were: acid concentration, reaction temperature, stirring speed, liquid-to-solid ratio, and particle size of sample. Table 3 shows the values of the five independent variables used in batch experiments to determine the response variations in the extraction percentage of Pt, Pd and Rh.

In order to identify the significance of the main and interactive effects of the variables combination, as it affects the extraction yield, data obtained from leaching experiments were statistically analysed using Fisher's ANOVA methodology. The results of regression analysis on reduced cubic model are given in Table 4. Values of “Prob>F” less than 0.0500 indicates high significant regression at 95% confidence level. According to the F- and p-values, concentration of HCl, temperature, and liquid-to-solid ratio were found more effective on the extraction of Pt, Pd, and Rh in the aqua regia leaching process. Figure 1 displays the correspondence between experimental and predicted values of PGM recovery yields. Fits showing good linear agreement between both values confirming the robustness of the model. In addition, the reliability of the model was checked by the coefficient of determination R². Predicted R² (Pt:0.9818, Pd:0.9860, Rh:0.9903) and adjusted R² (Pt:0.9111, Pd:0.9314, Rh:0.9645) values are close to one, which indicates that variables are highly correlated. In addition, experimental and predicted values are reasonably close to each other, standard deviation for Pt, Pd and Rh was 6.75, 4.52 and 3.91, respectively, indicating that the model fits the experimental data well.

According to the parameter values estimated from 45 batch experiments and statistical analysis (Tables 3 and 4), an empirical relationship between response and tested variable in coded units was obtained for each PGM (Equations (1)-(3)) by response surface methodology (RSM), as follows:

\[
\text{Pt}(\%) = 37.96 + 12.69A + 9.69B + 16.42E + 3.95D^2
\]

\[
\text{Pd}(\%) = 63.65 + 12.93A + 12.37B + 11.30E - 3.86A^2 - 3.80B^2 - 6.82AB^2
\]

\[
\text{Rh}(\%) = 26.21 + 16.07A + 9.19B + 11.08E + 3.47AE
\]

where Pt(%), Pd(%) and Rh(%) is the extraction yield percentage of PGM; A, B, D and E are the coded values of variables; concentration of hydrochloric acid in molL⁻¹ (A), temperature in °C (B), particle size in µm (D), and liquid-to-solid ratio in mLg⁻¹ (E). Particle size does not have an important effect on the extraction of Pd and Rh (Equations (2) and (3)), while it has only a quadratic effect for Pt (Equation 1). All three empirical models show that interactions between variables have no significant effects. Only in Equation (3) interaction with small positive effect between concentration of hydrochloric acid (A) and liquid-to-solid ratio (E) for Rh recovery is observed.

Table 3. Experimental design matrix for Pt, Pd and Rh extraction. A=HCl, B=temperature, C=stirring speed, D=particle size and E=liquid-to-solid ratio.

| Trial | Run | A (M) | B (°C) | C (rpm) | D (µm) | E (mLg⁻¹) | Pt (%) | Pd (%) | Rh (%) |
|-------|-----|-------|--------|---------|--------|-----------|--------|--------|--------|
| 1     | 6   | 7.50  | 39.0   | 164     | 64     | 3.60      | 63.26  | 71.83  | 57.44  |
| 2     | 19  | 11.50 | 39.0   | 164     | 64     | 3.60      | 29.97  | 56.51  | 22.76  |
| 3     | 41  | 7.50  | 76.0   | 164     | 64     | 3.60      | 46.64  | 72.78  | 33.39  |
| # | 1 | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9   | 10  | 11  | 12  | 13  | 14  | 15  | 16  | 17  | 18  | 19  | 20  | 21  | 22  | 23  | 24  | 25  | 26  | 27  | 28  | 29  | 30  | 31  | 32  | 33  | 34  | 35  | 36  | 37  | 38  | 39  | 40  | 41  | 42  | 43  | 44  | 45  |
|---|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 4 | 4 | 11.50 | 76.0 | 164 | 64 | 3.60 | 62.74 | 76.39 | 41.88 |
| 5 | 10 | 7.50 | 39.0 | 337 | 64 | 3.60 | 44.69 | 67.18 | 29.19 |
| 6 | 5 | 11.50 | 39.0 | 337 | 64 | 3.60 | 38.71 | 46.49 | 26.71 |
| 7 | 36 | 7.50 | 76.0 | 337 | 64 | 3.60 | 71.47 | 65.98 | 44.19 |
| 8 | 15 | 11.50 | 76.0 | 337 | 64 | 3.60 | 59.66 | 70.57 | 30.61 |
| 9 | 7 | 7.50 | 39.0 | 164 | 90 | 3.60 | 56.71 | 65.15 | 44.52 |
| 10 | 3 | 11.5 | 39.0 | 164 | 90 | 3.60 | 60.41 | 70.6 | 29.21 |
| 11 | 29 | 7.50 | 76.0 | 164 | 95 | 3.60 | 46.72 | 69.19 | 32.91 |
| 12 | 14 | 11.5 | 76.0 | 164 | 95 | 3.60 | 90.09 | 84.63 | 76.97 |
| 13 | 18 | 7.50 | 39.0 | 337 | 95 | 3.60 | 59.08 | 74.98 | 29.29 |
| 14 | 33 | 11.5 | 39.0 | 337 | 95 | 3.60 | 88.39 | 89.27 | 62.12 |
| 15 | 11 | 7.50 | 76.0 | 337 | 95 | 3.60 | 25.32 | 40.96 | 21.69 |
| 16 | 24 | 11.5 | 76.0 | 337 | 95 | 3.60 | 57.55 | 65.09 | 28.09 |
| 17 | 44 | 7.50 | 39.0 | 164 | 64 | 8.30 | 42.13 | 51.89 | 11.48 |
| 18 | 32 | 11.5 | 39.0 | 164 | 64 | 8.30 | 10.14 | 37.84 | 3.77 |
| 19 | 31 | 7.50 | 76.0 | 164 | 64 | 8.30 | 58.1 | 60.62 | 44.41 |
| 20 | 38 | 11.5 | 76.0 | 164 | 64 | 8.30 | 38.91 | 56.39 | 10.29 |
| 21 | 16 | 7.50 | 39.0 | 337 | 64 | 8.30 | 91.56 | 94.61 | 63.19 |
| 22 | 22 | 11.5 | 39.0 | 337 | 64 | 8.30 | 44.66 | 67.25 | 29.22 |
| 23 | 25 | 7.50 | 76.0 | 337 | 64 | 8.30 | 87.33 | 79.22 | 60.93 |
| 24 | 30 | 11.5 | 76.0 | 337 | 64 | 8.30 | 55.11 | 54.9 | 43.32 |
| 25 | 35 | 7.50 | 39.0 | 164 | 95 | 8.30 | 9.65 | 30.54 | 4.98 |
| 26 | 13 | 11.5 | 39.0 | 164 | 95 | 8.30 | 44.69 | 67.18 | 29.19 |
| 27 | 8 | 7.50 | 76.0 | 164 | 95 | 8.30 | 36.39 | 36.6 | 25.54 |
| 28 | 37 | 11.5 | 76.0 | 164 | 95 | 8.30 | 43.61 | 61.83 | 12.56 |
| 29 | 2 | 7.50 | 39.0 | 337 | 95 | 8.30 | 40.68 | 59.21 | 26.2 |
| 30 | 23 | 12.00 | 39.0 | 337 | 95 | 8.30 | 52.24 | 73.2 | 36.67 |
| 31 | 20 | 7.50 | 76.0 | 337 | 95 | 8.30 | 27.05 | 51.11 | 21.69 |
| 32 | 42 | 11.5 | 76.0 | 337 | 95 | 8.30 | 28.46 | 46.53 | 21.69 |
| 33 | 1 | 6.00 | 57.0 | 250 | 75 | 6.00 | 40.73 | 56.48 | 11.48 |
| 34 | 17 | 13.00 | 57.0 | 250 | 75 | 6.00 | 11.74 | 37.84 | 4.98 |
| 35 | 45 | 9.50 | 25.0 | 250 | 75 | 6.00 | 19.5 | 27.24 | 2.03 |
| 36 | 43 | 9.50 | 90.0 | 250 | 75 | 6.00 | 39.65 | 42.01 | 26.62 |
| 37 | 39 | 9.50 | 57.0 | 100 | 75 | 6.00 | 7.18 | 22.39 | 2.75 |
| 38 | 28 | 9.50 | 57.0 | 400 | 75 | 6.00 | 68.49 | 60.59 | 43.09 |
| 39 | 26 | 9.50 | 57.0 | 250 | 53 | 6.00 | 8.48 | 32.43 | 3.89 |
| 40 | 21 | 9.50 | 57.0 | 250 | 106 | 6.00 | 7.11 | 37.41 | 3.68 |
| 41 | 27 | 9.50 | 57.0 | 250 | 75 | 2.00 | 73.19 | 76.24 | 45.39 |
| 42 | 9 | 9.50 | 57.0 | 250 | 75 | 10.00 | 41.54 | 41.95 | 27.77 |
| 43 | 34 | 9.50 | 57.0 | 250 | 75 | 6.00 | 42.74 | 60.97 | 23.81 |
| 44 | 12 | 9.50 | 57.0 | 250 | 75 | 6.00 | 62.39 | 81.02 | 30.35 |
| 45 | 40 | 9.50 | 57.0 | 250 | 75 | 6.00 | 70.56 | 70.59 | 44.29 |
Table 4. Analysis of variance (ANOVA) for the reduced cubic model.

| Source | Sum of Squares | df* | Mean Square | F Value | p-value |
|--------|----------------|-----|-------------|---------|---------|
|        | Pt  | Pd  | Rh | Pt  | Pd  | Rh | Pt  | Pd  | Rh | Pt  | Pd  | Rh |
| Model term | 22156.3 | 12904.2 | 14098.9 | 1 | 1 | 1 | 633.0 | 368.7 | 402.8 | 13.9 | 18.1 | 26.4 | 0.0001 | < 0.0001 | < 0.0001 |
| A     | 957.5 | 994.1 | 1535.1 | 1 | 1 | 1 | 957.5 | 994.1 | 1535.1 | 21.0 | 48.7 | 100.5 | 0.0013 | < 0.0001 | < 0.0001 |
| B     | 907.0 | 909.9 | 502.1 | 1 | 1 | 1 | 907.0 | 909.9 | 502.1 | 19.9 | 44.6 | 32.9 | 0.0016 | < 0.0001 | 0.0003 |
| C     | 17.8 | 92.1 | 25.8 | 1 | 1 | 1 | 17.8 | 92.1 | 25.8 | 0.4 | 4.5 | 1.7 | 0.548 | 0.0626 | 0.2256 |
| D     | 7.9 | 33.8 | 41.4 | 1 | 1 | 1 | 7.9 | 33.8 | 41.4 | 0.2 | 1.7 | 2.7 | 0.6866 | 0.2302 | 0.1341 |
| E     | 1603.5 | 759.7 | 729.6 | 1 | 1 | 1 | 1603.5 | 759.7 | 729.6 | 35.2 | 37.3 | 47.8 | 0.0002 | 0.0002 | < 0.0001 |
| AE    | 0.0 | 3.2 | 386.0 | 1 | 1 | 1 | 0.0 | 3.2 | 386.0 | 0.0 | 0.2 | 25.3 | 0.9744 | 0.6998 | 0.0007 |
| A²    | 140.9 | 263.8 | 65.8 | 1 | 1 | 1 | 140.9 | 263.8 | 65.8 | 3.1 | 12.9 | 4.3 | 0.1126 | 0.0058 | 0.0678 |
| B²    | 8.3 | 167.4 | 20.2 | 1 | 1 | 1 | 8.3 | 167.4 | 20.2 | 0.2 | 8.2 | 1.3 | 0.6788 | 0.0186 | 0.2802 |
| C²    | 227.8 | 49.5 | 67.2 | 1 | 1 | 1 | 227.8 | 49.5 | 67.2 | 5.0 | 2.4 | 4.4 | 0.0522 | 0.1536 | 0.0654 |
| D²    | 275.8 | 33.0 | 38.0 | 1 | 1 | 1 | 275.8 | 33.0 | 38.0 | 6.0 | 1.6 | 2.5 | 0.0362 | 0.2354 | 0.1491 |
| E²    | 11.9 | 33.9 | 3.0 | 1 | 1 | 1 | 11.9 | 33.9 | 3.0 | 0.3 | 1.7 | 0.2 | 0.622 | 0.2292 | 0.6693 |
| AB²   | 22.5 | 233.2 | 24.3 | 1 | 1 | 1 | 22.5 | 233.2 | 24.3 | 0.5 | 11.4 | 1.6 | 0.5 | 0.0081 | 0.239 |
| Residual | 410.3 | 183.6 | 137.5 | 9 | 9 | 9 | 45.6 | 20.4 | 15.3 | --- | --- | --- | --- | --- | --- |

*Degrees of freedom.

*Obtained from experimental error estimated by the 3 replicates at central point.

Figure 1. Predicted vs experimental values of PGM extraction.
Metal dissolution depends more on the interaction between hydrochloric acid concentration and temperature rather than on other parameters. In this way, main effects of interactive relationship between independent variables on the PGM extraction yields are displayed in Figure 2. It can be observed that, in the range from 9.5M to 11.5M HCl, an increase in the temperature from 38.65°C to 76.35°C improves the extraction of Pt, Pd and Rh above about 30%. Consequently, at a certain temperature, an increase in the concentration of HCl enhances the extraction of metals. In the case of Pt extraction, increasing the concentration of HCl and liquid-to-solid ratio above 9.5M and 6.5mLg\(^{-1}\), respectively, has a positive effect on the leaching efficiency (data not shown); nevertheless, to maximize extraction of all of the metals simultaneously, the optimized values of the whole process could be different from that of a single process. Thus, optimum conditions have been found to be: 11.3M HCl, temperature of 70°C, liquid-to-solid ratio of 9.8mLg\(^{-1}\), stirring speed of 339rpm and particle size less than 94 µm. Under these conditions the rate of simultaneous PGM metals leaching reached to 93.9%, 95.01% and 64.5% for Pt, Pd, and Rh, respectively. Thereby, Pt and Pd were more easily leached than Rh from spent catalyst.

![Figure 2](image.png)

**Figure 2.** Response surface and contour plots for the effect of temperature (°C) and concentration of HCl (molL\(^{-1}\)) on the (a) platinum, (b) palladium and (c) rhodium extraction.

### 3.3. Comparison of different oxidizing agents in chloride media for PGM recovery

Dissolution of Pt, Pd and Rh is facilitated by the presence of chloride ions in acid media owing to formation of strongly complexed PGM ions [9,10]. Thus, in order to ensure a fair comparison between actions of different oxidizing agents, and evaluate the aqua regia efficiency, a series of leaching tests were performed under similar experimental conditions (Table 5). In all these experiments, the optimum parameters have been reported in literature [3,11]. Because for complexes the extent of formation is only depending on ligand concentration and the medium acidity, and considering that leaching systems contains high levels of dissolved chloride ions (11.5M Cl\(^-\) in all leach solutions) in strong acid media, it is expected that coordinatively saturated metal-ligand complex chlorides PtCl\(_6^{2-}\), PdCl\(_6^{2-}\) and RhCl\(_6^{3-}\) are formed [10,12,13].
Comparison of the effectiveness of different leaching systems are shown in Figure 3. Adding sulfuric acid to aqua regia (optimum conditions obtained above) and HCl/H$_2$O$_2$ mixture (leaching systems A and B, Table 5), ruthenium extraction increased significantly, by more than half (Figure 3(a),(b)). In contrast, no appreciable changes in Pt or Pd extraction was observed. On other hand, increasing the oxidizing power from the system B to system A leads to a slight increase of Pt and Rh extraction, and the opposite is true for Pd. Thereby, in leaching system A (18M H$_2$SO$_4$) the dissolution of Pt, Pd and Rh reached 94.08%, 94.53% and 89.95% (Figure 3(a)), while system B (18M H$_2$SO$_4$) achieved 89.22%, 95.71% and 83.02%, respectively (Figure 3(b)). It is noteworthy that the highest extraction yields were obtained for leaching systems A and B. Indeed, oxidants and reactive intermediate species generated in leaching systems A (nitrate, nitrosyl ion and chlorine gas) and B (hydrogen peroxide, atomic oxygen and chlorine gas) are strong oxidants. In this way, PGM can be passivated by these chemical species, however, chloride ions are strongly chemisorbed on PGM surface.

Specific adsorption of chloride ions on the PGM can inhibit irreversible oxide formation [9], hence, dissolution process arises through charge transfer coupled with complexation of Cl$^-$ at PGM/solution interface. Increasing the concentration of sodium hypochlorite, a strong oxidizing agent, has no noticeable effect on the recovery of any metal (Figure 3(c)). In addition, the lowest recoveries were obtained with this lixiviant system, below 65%. This is explained because sodium hypochlorite exhibits chemical instability in acidic solutions, and although sodium hypochlorite generates chlorine gas, another strong oxidizing agent, their solubility decreased with the increase of temperature (leach tests were carried out at a temperature of 80°C) [14]. Copper(II) in system D is a weak oxidizing agent compared to aforementioned oxidants; however, extractions are larger than that obtained in system C with sodium hypochlorite/chlorine gas. The effect of increasing Cu(II) concentration on PGM extraction is marginal. Certainly, the lowest copper concentration here used was found as the optimum value to obtain the highest PGM recovery in a recent study [11].

### Table 5. Compositions for each leach test.

| Lixiviant system | Reagent concentration (M) |
|------------------|---------------------------|
|                  | HNO$_3$ | H$_2$SO$_4$ | H$_2$O$_2$ (vol) | H$_2$SO$_4$ |
| **A**            | 3.85    | 0           | 12.0             | 18.0        |
| **B**            | 110     | 0           | 12.0             | 18.0        |
| **C**            | 3.0     | 5.0         | 8.0              | Cu(II)      |
| **D**            | 0.3     | 0.5         |                  |             |
Figure 3. PGM extraction yields as a function of leach solution composition (Table 5): (a) A, (b) B, (c) C and (d) D. Conditions used in all leaching tests: 11.5M HCl, stirring 400rpm, temperature 80°C, particle size -63 µm, solution/solid ratio 8.5mLg^{-1} and time 240min.

4. Conclusions
In order to obtain optimum simultaneous extraction of Pt, Pd and Rh from catalytic converters of spent exhaust systems, a series of leaching tests were carried out to investigate the key variables in evaluating leaching behaviour by response surface methodology. Mineral acid concentrations and temperature were the most influential factors in the PGM leaching. At an optimum composition of the leach solution, aqua regia/sulfuric acid mixture was the best medium combination for PGM dissolution at almost 95% of Pt and Pd extraction, and yield of about 90% for Rh. The optimum leaching conditions for 4h were: leaching temperature 70°C, ratio of liquid to solid 9.8, stirring speed 337 rpm, particle size less than 94µm, acids concentration 11.3M HCl and 3.4 M HNO₃ (3 HCl:1 HNO₃ volume ratio). Strong acidification of either aqua regia or piranha in chloride medium is beneficial to enhance the PGM extraction performance. Soluble chloride complexes of PGM prevent the formation of insoluble products that could passivate the metal surface.

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