

## **SEM Characterization Ni-Mo Catalyst Synthesized by Mechanical Alloying**

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For many years, some transition metals such as Ni, Mo, Fe and Co have been broadly used as precursor materials for the elaboration of catalysts.

Petrochemical industries have been the target of much research looking forward to optimize the conventional processing of crude oil refinement. Ni and Mo-based catalyst had proved excellent catalytic activity.

Ni (99.9 % pure, -200 mesh in size), Mo (99.5%, -200 mesh) and Al (99.9%, -200 mesh) crystalline metal powders were used as raw materials. Nominal composition was set to  $(\text{Ni}_{75}\text{Mo}_{25})_{50}\text{Al}_{50}$  at %. The mechanical alloying (MA) experimental runs were performed at room temperature in a commercial high-energy laboratory ball mill (Simoloyer). The milling time intervals were decided to be 9, 18 and 36 h based on previous results. The milling media to powder weight ratio was maintained constant to 20 to 1 for all experiments.

Microscopy (SEM) was conducted in a JEOL JSM 5800 LV supplied with EDS spectrometer. Surface area (SA) determination was carried out in a Quantachrome equipment model Autosorb B-1, using nitrogen gas as the adsorbent element. The samples were degassed at 573 K under vacuum for 2 h before analysis.

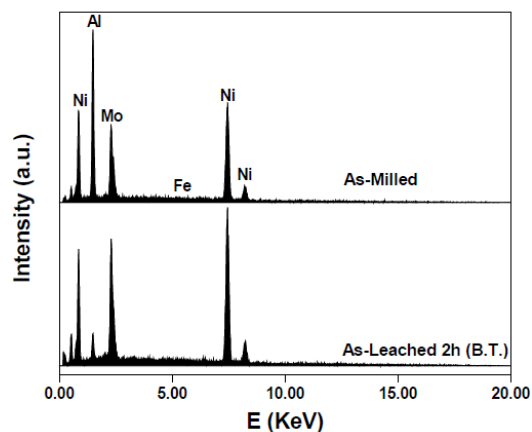
Raney-type Ni-Mo-based catalysts were produced using mechanically alloyed Ni-Mo-Al powders, followed by an alkaline leaching process. Aluminum is removed in the latter process to induce large SA values. Synthesized catalysts were found

highly actives due to their nanocrystalline nature synergized by its large surface area. The catalysts produced by this route presented good selectivity for the hydrodesulphurization (HDS) reaction in dibenzothiophene (DBT). Catalysts showed larger reaction constant values than its commercial counterparts. The temperature and leaching time have an important effect in the catalytic activity.

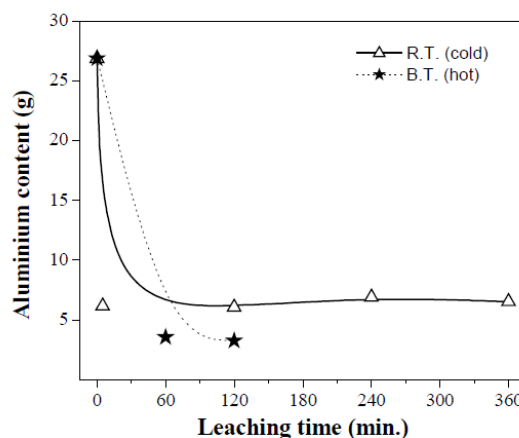
Figure 1 shows EDS spectra from as-milled and as-leached samples. It is evident that the intensity of typical Al peaks decrease significantly after the leaching treatment; meanwhile, common diffracted peaks from Ni and Mo are still in the same ratio. This confirms that the chemical dissolution occurred by using alkaline solution is selective for Al. Additionally, the contamination (Fe from milling media wear) levels were kept under 0.5 at %.

Figure 2 shows the variation of Al content as a function of leaching time. A higher Al dissolution was reached at short processing times. Figure 3 shows a representative view of the powders microstructure in the as-milled condition (a), after 1h (b) and 2h (c) of leaching at boiling temperature (BT).

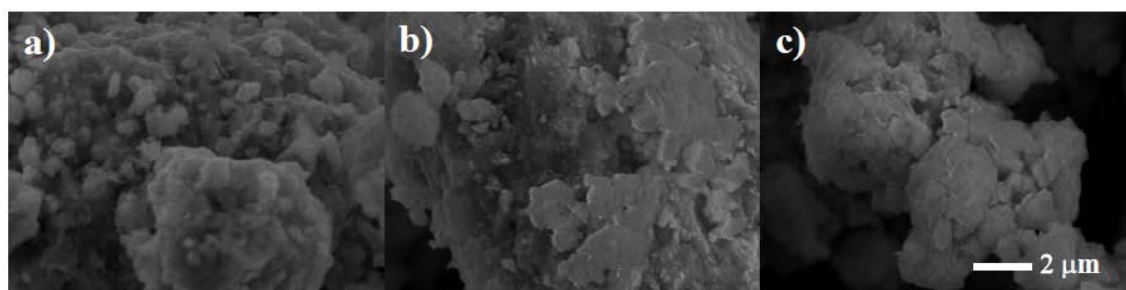
Table I resumes the experimental results of this work. This Table presents the Al content before and after leaching treatment; as well as the SA values found after Al dissolution.



**Figure 1.-** EDS patterns of milled and leached  $(\text{Ni}_{75}\text{Mo}_{25})_{50}\text{Al}_{50}$  samples.



**Figure 2.-** Aluminum content found in the milled powders in function of leaching time.



**Figure 3.-** SEM micrographs of  $(\text{Ni}_{75}\text{Mo}_{25})_{50}\text{Al}_{50}$  powders prepared by MA and leaching a) As-milled, b) 1 h leaching and c) 2 h leaching.

**Table I.-** Al concentration values of some samples after a mass balance correction of elementary analysis obtained by EDS and other important parameters of obtained catalysts.

SAMPLE	Leaching Temperature	Al Contents (basis 100g)	% Al Removed	SA [ $\text{m}^2/\text{g}$ ]
COMMERCIAL	-----	-----	-----	> 150
As-milled	-----	28.40	-----	3.08
60 min.	BT	3.56	87.46	87.02
120 min.	BT	3.28	88.45	100.58
5 min.	RT	6.18	78.24	55.72
120 min.	RT	6.08	78.59	71.80
240 min.	RT	6.90	75.70	79.70
360 min.	RT	6.55	76.94	36.86