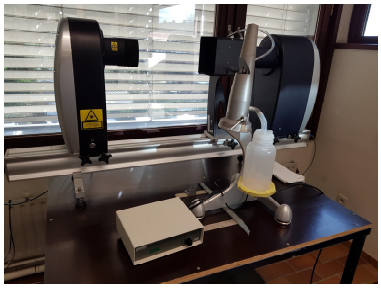
Heterogeneous catalyst optimisation by particle size determination

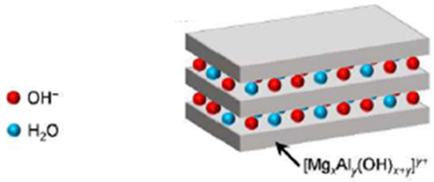
Summary:

The efficiency of heterogeneous catalysts depends on the active sites on the surface. Particle size control is essential to realize high activity.
BET surface area gives an indication on surface area. However it is a difficult parameter to understand effects occurring during size reduction. It was observed that milling, using a mortar, of a catalyst did not increase the catalytic activity, although the powder was clearly finer. Laser diffraction allowed to understand mechanisms occurring during milling and is hence a important additional tool to be used.

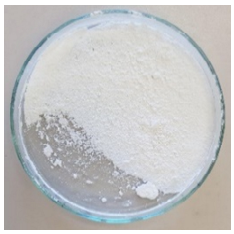


Experimental part:

Hydrotalcite[1] can be used as source of OH^- groups for base catalysed reactions such as oxidation by the hydroperoxide anion.
The number of active sites in the hydrotalcite is important to estimate the catalytic efficiency.
The milling of hyotalcite was done using an automatic mortar (Pulverisette by Fritsch) .
The change in particle size was measured by laser diffraction[2].

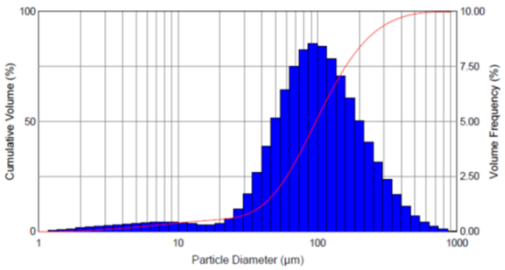


$$H_2O_2 + OH^- \rightarrow OOH^- + H_2O$$

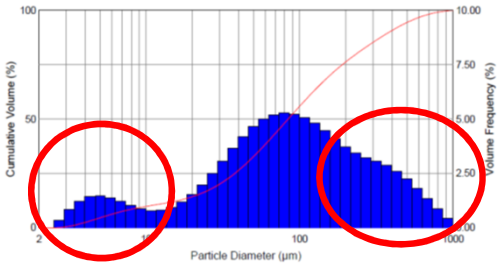


Results and discussion:

Effect of size reduction by milling as observed by laser diffraction.



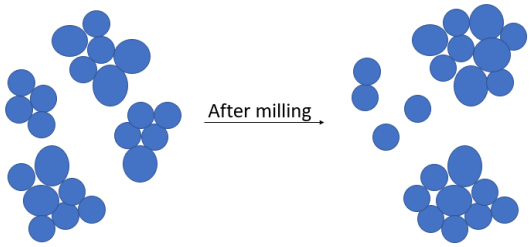
After milling
2 passes



The main parameters obtained are reported in the table below:

Sample	Dv(10) (µm)	Dv(50) (µm)	Dv(90) (µm)	D[3][2] (µm)	Span
Before milling	36,27	97,22	250,9	44,56	2,207
After milling	10,29	84,09	383,1	29,26	4,434

The total distribution shows a reduction of Dv(10), Dv(50) and D[3][2] and an increase of the span and of the Dv(90) volume.
Since the catalytic activity did not change, BET surface should have remaining identical. Analysis of the solid indicate that milling by a mortar did not affect the primary particle size 2-4 um.
The powder was de-agglomerated by the milling (increase of low size fraction) but at the same time a re-agglomeration occurred (increase of >100 um agglomerates).
This process was confirmed by microscope. Conclusion: laser diffraction can be used to get more information on milling process. In the actual case, further research will be done to stabilise the de-agglomeration to improve surface accessability.



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[1] J.Mater.Chem. A 2017, 5, 16200-1611
[2] https://www.malvernpanalytical.com/en/products/product-range/spraytec

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