## TREATMENT OF RESIDUARY ACIDS BY MICROWAVES

## Jean-Paul BERNARD\*, General Manager

Company SAIREM, 12 Porte du Grand Lyon, 01707 Neyron Cedex, France, phone : +33 4 72 01 81 60, fax : +33 4 72 01 81 79

Many chemical syntheses take place in an acid medium (sulphuric or/ and nitric): the recovery of the final product requires a specific treatment of the reactional medium (separational or extractional ...) before the acid is reclaimed in the process.

In the case of nitro-glycerine (Ngl) synthesis, the treatment of the residuary acids, which come out of the synthesis workshop and contain 2,5 % of the final product, must be carried out with great care. In effect, the decant nitro-glycerine is then supernatant and may crystallise out, hence some risks of explosion.

The conventional method for the treatment of these acids consists in injecting water vapour (vapour temperature : between  $130^{\circ}C$  and  $150^{\circ}C$ ) into the column fed at the foot with the solution to be treated.

The heat pick-up provokes a heating of the medium and triggers off the following reaction :

 $Ngl + 5HNO_3 \Rightarrow 6NO_2 + 2 NO + CO + 5H_2O$  $150^{\circ}C$ 

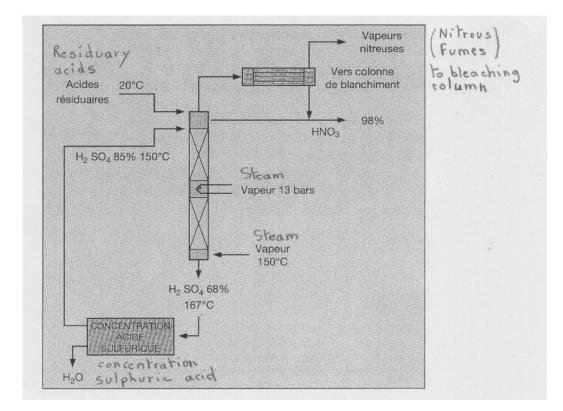


Figure 1. Diagram of the classical process.

This treatment presents many disadvantages:

- very long heating time (several hours) as very high temperature gradients have to be avoided
- little flexibility
- high treatment cost
- dilution of residuary acids, therefore a need for a reconcentration phase for the solution to be treated before it is reclaimed in the process.

The national company of powders and explosives (SNPE France Angoulême) is moving towards a new technology: microwave.

The SNPE has listed a new pilot for the treatment of acids. The tests results have brought to light the advantages of microwave.

The main asset of microwave is a heating without contact, hence no risk of corrosion. The other assets are:

- no dilution of the acid recovered
- a better expertise in the process (in the type of temperature)
- a lower power consumption in comparison with the traditional system.

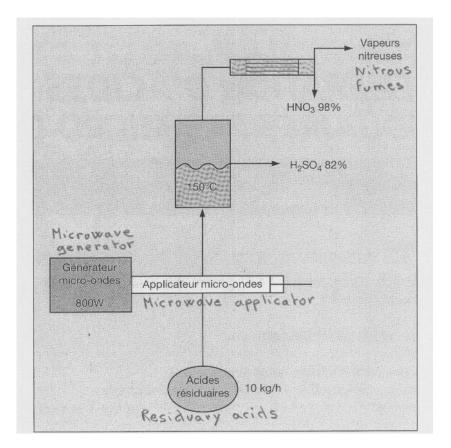


Figure 2. Diagram of the microwave process.

The SNPE has built up two industrial units (1 in France and 1 in Belgium). Each unit allows to treat 150 kg/ hour of residuary acids. The microwave power needed is equal to approximately 7.2 kg.

The acid is pre-heated at  $65^{\circ}$ C /  $70^{\circ}$ C. The pre-heating is carried out by a thermic exchanger which recovers the calories of the residuary acid treated by microwave ( acids temperature :  $120^{\circ}$ C/  $130^{\circ}$ C ). This allows to save between 2 kW and 3 kW of microwave power.

Table 1. Comparison of the Investment Costs and the Running Costs.

TREATMENT	TRADITIONAL	MICROWAVE
INVESTMENT COST	808 000 Euros	795 000 Euros
<b>RUNNING COST</b>	8.08 Euros / ton of raw acid	3.81 Euros / ton of raw acid

For a load of 150 kg / h:

Energy prices 13.00 Euros / ton of vapour 0.04 Euros / kW per hour

## PROCESS

<u>Mix</u> Glycerin + Sulfuric Acid + Nitric ACid Creation Nitroglycerine + water (water remains with the sulfuric) Sulfuric Acid

## CONCLUSION

The treatment of residuary acids by microwave presents many advantages, essentially in case of effluents containing organic impurities which can be decomposed easily. This enables to cater for a double objective: on the one hand, this enables to reclaim some raw material in the process, and on the other hand, this enables to fight against environment pollution.