Sulphuric Acid Concentration

In the early seventies, Worley Chemetics (formerly, Chemetics®) engineers set out to improve the sulphuric acid concentration process by building upon their knowledge and experience with Nitric Acid Concentration.

Using **modern, reliable materials** such as glass lined steel, PTFE, Tantalum and Zirconium, the engineers at Worley Chemetics developed **the patented vacuum evaporation process that still forms the heart of our technology today**. This process is characterized by the extensive use of gravity flow to transfer hot acid between the various process steps and by the use of natural circulation thermosyphon loops for the acid evaporators.

These natural circulation evaporators allow high heat transfer rates, large turndown, minimize fouling and avoid the use of pumps in hot acid service which **improves both safety and reliability**. Multiple stages are used for large capacities or for large increases in concentration.

In 1975, after extensive pilot plant work, the first modern Worley Chemetics Sulphuric Acid Concentration plant was designed and built for Air Products DNT facility in Texas, where it continues to operate today. Since that time, the design and technology have continued to improve and **more than 70 spent acid plants** have been delivered to satisfied clients around the globe. Completed plants range in capacity from **25 to 2200 MTPD** and concentrate sulphuric acid from a starting concentration as low as 7 wt% to product concentrations between 83-96 wt% sulphuric acid.

Although the Worley Chemetics Evaporator System forms the heart of every process, it is very important to incorporate into each plant **the appropriate pretreatment** of the spent acid to remove (and recover) impurities and to minimize effluent. Worley Chemetics has developed several additional process option that can be used to provide a custom made process for each spent acid. Examples are **hydrolysis or stripping processes** to remove organics, **fractionation columns** to concentration and recover valuable by-products, and **NO_x absorption systems** to clean vent gases prior to discharge.

Worley Chemetics carries out **extensive safety reviews** and operates a testing facility to ensure that optimum conditions are selected for each spent acid. This approach has resulted in **safe and reliable plants** with availabilities well above 95%.

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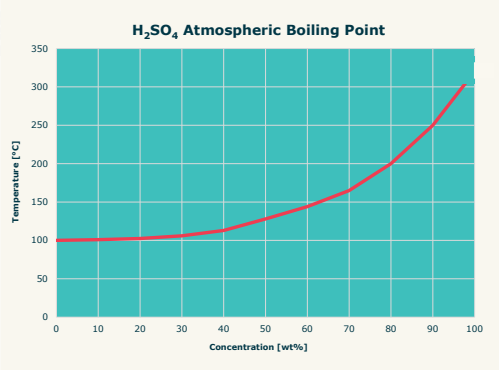
BACKGROUND

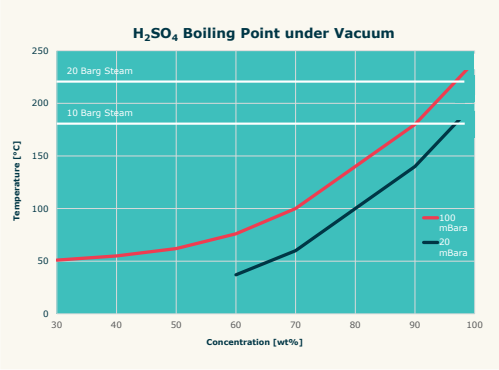
The concentration of sulphuric acid provides a number of technical challenges/choices that must be carefully managed in order to create an economic, reliable process.

Sulphuric acid can be concentrated by boiling the solutions as water evaporates preferentially. The graph below shows the atmospheric boiling point of sulphuric acid at various concentrations:

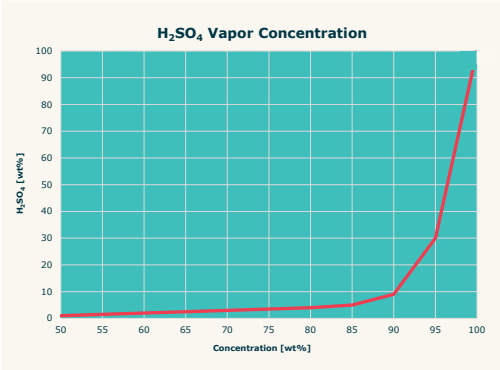


It is clear that high temperatures are required to obtain concentrated acid. When the required temperature exceeds the temperature of the available heat source (normally steam) and/or the temperature limits (approximately 200°C) of the available materials of construction, it is necessary to carry out the concentration under vacuum. The vacuum levels used in practice are further constraint by the desire to condense the water that has been removed using normal cooling water resulting in a practical lower operating limit of approximately 100 mBara.

As can be seen below, using 10 Barg steam, it is possible to concentrate up to ~85 wt% at an operating pressure of 100 mBara. If 20 Barg steam is used, the maximum possible concentration at 100 mBara pressure increases to ~90 wt%. For even higher concentrations it is necessary to operate at a lower pressure and the use of chilled water to condense the overhead vapours is required.



At high concentrations a further complicating factor comes into play. As the acid concentration increases, the vapors removed from the boiling liquid consist not only of water vapor, but also contain an increasing amount of sulphuric acid vapor. The sulphuric acid vapor concentration increases until the azeotrope is reached at ~98.3 wt% H_2SO_4. The acid concentration in the vapor is shown in the graph below:



Based on this graph it can be concluded that acid concentrations up to approximately 89wt% are possible with acceptable (<2.5 wt%) acid losses to the overhead and no special precautions have to be taken to prevent corrosion of the overheads condensing system or recover the acid lost from the system.

At higher concentrations additional process equipment is required to remove and recover the sulphuric acid from the evaporator overheads. Generally a packed column is used to contact the vapours with a dilute sulphuric acid liquid stream. This causes the sulphuric acid vapours to be selectively removed after which the water vapour can be condensed in the overheads condensing system.

Due to the presence of the azeotrope at 98.3 wt% H_2SO_4 concentration it is not practical to concentrate to a product concentration that exceeds 97wt% H_2SO_4. If a higher concentration is required, it is possible to fortify the acid further using either SO_3 gas or Oleum.

Worley Chemetics has over 40 years experience concentrating sulphuric acid from a wide range of sources. Around the world, acid concentrators designed by Worley Chemetics concentrate sulphuric acid from starting concentrations as low as 10 wt% to product concentrations exceeding 96 wt% – we are ready to apply our knowledge and experience to meet any sulphuric acid concentration requirements.