

# The Whatmans and Wove Paper

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## In Defence of Alum - 1. The early history

Although alum is not specifically relevant to the development of wove paper, this chemical was used by the Whatmans in the production of their celebrated high quality papers, made during the 18th C. Provided papers like these have not been exposed to a hostile environment during the two hundred and fifty years following their manufacture, we find that they are as robust as ever they were, and some much more so than many papers made today. Indeed the same applies to most other top quality papers made in this and other countries in the 16th and 17th Cs., as well as to Class I papers produced during the 19th and 20th Cs. And yet, nowadays, for some reason the presence of alum in paper is regarded as undesirable, even harmful. The questions posed by this are, does alum deserve this reputation? What is its function in papermaking? And, finally, are we absolutely clear what "alum" is?

In papermaking the distinction between "alum" and hydrated aluminium sulphate has become blurred during the transition from the one substance to the other over the last hundred years. In fact Labarre (Bib.22, 1937) defines it as a "term now used for aluminium sulphate". To help us answer these questions, a very condensed history of the chemical follows, with a description of the process used in its manufacture in England during the period covered by this book, for which due acknowledgment is made to Professor Singer's great monograph and to more recent work carried out by members of the Cleveland Industrial Archaeology Society; see Footnotes and Bibliography.

### 1. The early history of alum

The procurement of alum has been dubbed "The earliest Chemical Industry" by Professor Charles Singer in a monumental study of its occurrence and of its manufacture in the past (Bib.57). There are oblique references to alum, sometimes very obscure, dating back at least to two millennia B.C. But the certainty of its actual usage can only be proved by analysing and dating artefacts in which alum had been employed at some stage of their creation, undoubtedly an activity of even greater antiquity. It was used mainly for mordanting; for tanning and softening leather; for its alleged medicinal and cosmetic properties; and as an auxiliary agent in miscellaneous metal and glass finishes.

Alum occurs naturally in more than one form; but, where it was not readily available, it had to be manufactured. The processes involved ranged from relatively simple methods to extremely sophisticated ones. In principle the latter made use of certain sedimentary aluminium bearing rocks or shales which were converted by various means to yield aluminium sulphate, which, in turn, was converted into alum, usually as the ammonium or potassium double salt.[1]

The earliest known uses of alum in the Mediterranean area, and indeed anywhere, are referred to in Egyptian records of 2000 B.C. They do not identify "alum" specifically, but, as mentioned above, its presence can be deduced from the things for which it was used, both in its local application and from those found in other countries, e.g. descriptions in cuneiform or Mesopotamian tablets of the 8th C. B.C. By the 5th C. B.C. significant quantities of alum were being exported from Egypt to other countries in the Middle East.

The Ancients had discovered that alum possessed certain valuable qualities not found together in any other common substance: its extreme readiness to crystallize; the absence of colour as an indication of its purity (extremely important in dyeing); and its natural occurrence. Its sources were, therefore, identifiable with relative ease. Singer has pointed out, "that alum was obtainable pure was thus a technical accident and not the result of any considered rational plan or of any refined system of manufacture". The sophisticated methods of manufacture, referred to above, took millenia to evolve. The process that is of most interest to us in the present context, the one used in the alum industry of North Yorkshire, has been described as "a marvel of empirical industrial chemistry".[2]

The extension of the alum industry from Egypt to other regions is an extremely complicated subject, partly due to the linguistic problems it raises, ensuring that one is concerned with alum and not with another substance; and, more important, the confusing effects on trade of one empire after another being superseded in Asia Minor and Eastern Europe, from Persia in the east, through North Africa to Spain in the west, continuing from early periods right up to the Middle Ages. The Greek, Roman, Byzantine and Arabian civilizations were all involved, together with other sources of influence, penetrating this world from India and, by the silk route, from China. For detailed information on the effect they had on the supply and use of alum, reference must be made to Singer's monograph.

It was from all the sources mentioned above that the first methodical statements on the nature of alum were derived. The findings are set out in 8th-10th C. (A.D.) Arabian alchemical documents. By this time the Arabs had conquered the whole of North Africa and thus confronted, and even penetrated, the southern borders of Europe. Trade links between the two were strong and there was no shortage of European scholars who were able to read arabic and assimilate its learning. It might be noted at this juncture that it was just after this period that papermaking was undertaken for the first time by Europeans, albeit the use of alum in this activity lay sometime in the future. (See Introduction p.xxiv).

The demand for alum from countries to the north of the Mediterranean was an ancient one; but from the 10th-12th Cs. dyeing, and the consequent use of alum, had become increasingly monopolized by Jewish communities who had migrated to Italy and Provence from the Byzantine Empire and to Spain from Tunis, and who had yet another stronghold in Alexandria. The sources of supply were various. Some European countries favoured supplies from Sicily and the Aeolian islands; Spain had its own resources, with especially high quality coming from Castile; but the best qualities still came from the Middle East. The four Crusades of the 12th C., however, interrupted many of the established trade routes and aggravated the conflicts between Saladin and the declining Byzantine Empire, creating the embryonic elements of the dramatic changes that were to come. For one thing, the last crusade helped polarize the antagonism of the Roman and Eastern churches.

Meanwhile new agencies were affecting this situation. The Mongols sacked Baghdad in 1258 shattering Arabian culture at its source; the Turks were beginning to dominate the middle eastern scene; and, equally important for the future development of alum supplies, the City States of northern Italy were transforming and widening the market. They included, initially, the Venetian Republic, Genoa and Pisa. Pisa, however, soon succumbed to the expanding power of Florence. Genoa not only built up lasting trade links with north-western Europe, e.g. with important centres such as the Duchy of Burgundy and the extensive markets of the Hanseatic League in the Baltic, but it acted as a middleman, dyeing and finishing textiles imported from these centres and selling them to Sicily, Egypt and the Levant. Its sources of alum came via Alexandria; Bejaïa (Algeria), brought there by caravan from Lake Chad; Castile and Greece. Genoa was also to expand its sources of alum and trade into the Black Sea and off the eastern coast of Asia Minor, the latter especially vulnerable to embargoes or attack from the Turks.

In the longer term it was Venice that reigned supreme. Geographically she was at the cross roads of East-West trade routes, with access to central Europe to the North. Since the 9th C. Venice had been a subject of the Eastern Empire. With the decline of Byzantium, and its ultimate fall, she became the real ruler of this Empire, having steadily acquired important concessions in almost all parts of the Levant. She had powerful settlements in Constantinople, Cyprus and Crete. As a result she had access to innumerable sources of alum. To begin with she was not a manufacturing city, but served as a carrier of goods, including alum, to other parts of Europe. Later, she developed her own industries, some of which required alum. But, as Singer points out, "Venice remained to the end a merchant-state... maintaining mercantile relations with both the Christian and Moslem worlds". For details of the varying fortunes of these City States, and the rise of many other centres in Europe, the reader is once again referred to Singer's monograph.

After 1453 (the fall of Constantinople) major problems arose regarding future supplies of alum to meet the ever increasing demand. The Levant was now under the control of the Turks; Western Europe was facing a new situation. Before considering the implications of this change, it would be as well to describe, briefly, some of the mineral sources from which alum could be obtained.

As said earlier, alum occurs in small quantities naturally; otherwise it has to be manufactured from two principal sources, alunite (alum rock or alum stone) and alum shale (alum ore or alum schist). Small quantities of natural alum (K<sub>2</sub>SO<sub>4</sub>.Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.24H<sub>2</sub>O) could be found, as clearly recognizable crystals, in the Egyptian deserts (south-west of the Oasis, El Khârga). It was brought in by the Bedouin and shipped down the Nile to Alexandria. Another ancient source came from the Yemen. This, however, was crude aluminium sulphate and needed skilled treatment to convert it into ammonium alum. Urine was added to it, the solution boiled and the alum allowed to crystallize out of it on cooling. Sources of this kind were clearly limited.

In its manufacture, the same principle was used to make alum from both mineral sources referred to above, the main difference between the two processes being that with alunite, the effects of volcanic activity, and deposits associated with this, were responsible for taking care of the preparatory stages normally required for extracting soluble aluminium sulphate from alum shale.

Deposits of alunite (K<sub>2</sub>SO<sub>4</sub>.Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.2Al<sub>2</sub>(OH)<sub>6</sub>) were found not only in Asia Minor, but all over Italy, for example, close to sites of recent volcanic activity (Piomboino, Volterra, Pozzuoli near Naples, Elba and the Aeolian islands). Large quantities of trachyte found in this environment (formations of igneous rocks mainly composed of aluminium silicates) were converted into alunite by local discharges of sulphurous fumes or acid. Alunite was only a half-way stage to alum. It was necessary to separate excess alumina from it by roasting and lixiviating the ore. Roasting often made the alum rock porous, facilitating the extraction of the alum, which could then be further purified by boiling and recrystallization.

The sources of alunite, however, were limited to areas of active or extinct volcanic extrusion. Since the demand for alum was continually growing, other means of obtaining it were needed. The so-called alum shales provided the answer. Apart from accessibility and other economic factors, the main criteria for success in working them depended on the presence of aluminium salts, pyrites and carbonaceous material, such as oil, in the schists, as well as the availability of fuel, usually coal, for boiling and evaporating the extracts. The process will be described in some detail when the manufacture of alum in North Yorkshire is discussed later. There the production of alum flourished from the mid-17th to the mid-19th Cs. It is obvious that this period is two centuries later than the time of the crisis faced by Europe, referred to above.

Singer believed that the shale process had been known for a considerable period before the demise of Byzantium to the Turks. He cites, for example, a laboratory preparation of alum from shale carried out by the Arab, Razi, in the 13th C. However, he found no evidence that alum had been extracted from shale on an industrial scale before the 15th C. This occurred in central Europe and was first exploited in the Tyrol. The discovery would appear to have been accidental, resulting from the mining of pyritic shale, long practised in this area. An alum works was established not far from Innsbruck in the 15th C. It soon attracted the attention of prospectors from Venice, Verona and Saxony. The Tyrol itself never became an important source of alum, but the news spread quickly to other parts of central Europe, notably Bohemia, Silesia and adjoining districts, where by the mid-16th C. many alum works had been set up.

By this time alum ranked among the more precious cargoes that were traded across Europe, valuable enough for English privateers to seize ships carrying it and taking it to home ports, where it was sold to the growing *Cloth* industry in Britain (see Bib.2 Vol.I Chapter II p.63). By the late 15th C. most of the prime qualities of English Cloth were made from wool dyed before weaving. Other countries had also realized the importance of having a domestic supply of alum, which led to further exploitation of deposits of alum shale. By the late 16th C., for example, the manufacture of alum had been established at Liège, which became an important centre up to the first quarter of the 19th C., supplying France and many parts of central Europe with this commodity; although France too developed a domestic industry of its own at the beginning of the 16th C. For details of the further expansion of the shale alum industry, extending to Scandinavia, the Rhineland and other parts of Germany, the reader is referred once again to Singer.

What lay behind the sudden expansion and dissemination of this industry? Put in very simple terms, there were two main factors that brought this about; first, the military domination of Asia Minor and the Levant by the Turks, a situation which culminated in the majority of the traditional sources of alum for the West coming under their control. Second, social and political attitudes were altering dramatically in Europe at this time. The religious and economic climate was ripe to receive changes. The great trading centres had developed beyond their former boundaries; and there was ample scope now for entrepreneurs and prospectors.

Although trade links continued for a time between the West and the Levant, the fall of Constantinople and the Eastern Church eventually brought about the migration of skilled dyers, alum workers and others from east to west. Moreover, not content with conquering Byzantium, the Turks were preparing to invade Eastern Europe, putting the West on the defensive.[3]

A distinguished lawyer, Giovanni de Castro, (and friend of the Pope Pius II), who had experience of the dyeing and alum industries in and around Constantinople, recognized large quantities of alunite in the vicinity of Tolfa, a small town about 25 miles north of Rome and within the Papal States. In 1458 Giovanni had become commissary of the Apostolic revenues. He quickly realized that the huge flow of money from the West, that paid for the alum and dyeing, formerly practised in the East, could be reversed by his discovery, with the Pope dominating this trade in future.

Within five years Tolfa was producing alum of very high quality on an industrial scale. Genoa and Florence were the first major customers, with Venice following suit. Outside Italy the most important markets were the Flemish ports, thence to England, Northern France, the Hansa and Germany. However, this picture has been greatly simplified. The real state of affairs is far too complicated to describe here.[4] The Popes regarded the deposits at Tolfa as a divine gift and wanted profits to be devoted to the defence of Christendom with a further crusade against the Turks. But this idea was not universally accepted. Some countries, Venice in particular, continued to import Turkish alum. Threats of excommunication were ignored; the seeds of the Reformation had already taken root. The Popes tried in vain to enforce a monopoly, but were outmanoeuvred.

As we have seen above, new sources of alum, manufactured from shale, contributed to the decline of the output from Tolfa, assisted by the substantial quantities of alum still imported from Turkey and distributed by Venice. Over the next half century the Papacy fought back and Tolfa survived; but this time, as Singer puts it, "for chemical rather than spiritual reasons". Another factor that affected the balance in this trade was the decisive defeat of the Turks in 1571 at the Battle of Lepanto, the Turks never regaining their sea power.

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### Footnotes

[1] Other similar forms with astringent properties were known, such as iron, magnesium and manganese alums.

[2] Rayner & Hemingway (1974).

[3] The Turks controlled the mouth of the Adriatic and, in fact, were invading Italy, taking Otranto in 1480. This was recaptured a year later.

[4] Even Singer found the machinations of the Popes and entrepreneurs, such as Lorenzo Medici and Agostino Chigi, too complicated to resolve, coupled with the fact that many countries continued to buy their alum from the Turks, Venice being at the forefront in this trade.