Medieval Gunpowder Research Group

Saltpetre from India

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A Galathea 3 Project

Middelaldercentret, Nykøbing, Denmark

Introduction

One of the main aims of the Medieval Gunpowder Research Group has been to make and test gunpowder made from ingredients produced as closely as possible using methods of the medieval or early modern periods. We have managed to make our own charcoal in the traditional ways. We have also collected sulphur from the sites where it was mined in the medieval period and we are currently experimenting with different ways of refining it. But it is the saltpetre which is proving to be a problem and our attempts to make it have, so far, failed. In order to try to understand the problems involved and their solution the Group travelled to India to look at how saltpetre was produced there.

Saltpetre and India

When European explorers discovered the sea routes to India at the very end of the 15th century they found a culture and civilisation as old as their own and one which was as advanced, in many areas even more advanced. By the closing decades of the 16th century ships were regularly trading with the Indian sub-continent as well as with much of South East Asia – mostly in high value goods, such as spices, which brought the best returns on their investments. But spices were not the only commodity that they found and in India the European merchants discovered that saltpetre was being made – and made in large quantities. From the beginning of the 17th century, many European nations, mainly the Portuguese, Dutch and the English but also the Danes, French and Swedes organised their trade to the East and set up monopoly trading companies for the purpose. And from then on saltpetre was increasingly traded and exported to Europe. For example the English, from a level of just tens of tons in the 1620s, were exporting from India an average of around 500 tons per year from the 1660s on. By the 1740s the trade was approaching 1000 tons a year and peaked at over 2000 tons in 1742 and 1743. The export continued right down to the end of the 19th century – Marshall (1917: 57) states that in the middle of the 19th century the trade was over 30,000 tons but this dropped down to 18-20,000 by the beginning of the 20th century.

Because the manufacture of saltpetre continued well into the early years of the 20th century, there are a number of written and published accounts of the production of saltpetre, especially from the 19th century, which give many details of the methods used. These indicate that a process very similar to that used in Europe from the end of the 14th century was still being used right down to the 20th century in India. If we could find the remains of that process and study it we could gain new insights into the production of saltpetre from medieval and early modern times in Europe.

Study visit

The purpose of our visit to India was to try to locate any surviving remains and make a study of them. Early sources indicate that the areas of greatest production were the state of Bihar, especially around the capital, Patna, and the West Bengal area. However a Danish colleague of the Group who knows India well had information that saltpetre was being made in the area to the north east of Agra in Uttar Pradesh. Our itinerary was therefore put together with a view to exploring this area first and if we drew a blank, then to move on to the Patna area.

In addition the Group also wanted to try to investigate further a reported find of medieval gunpowder in 2002, in Jaisalmer in the western part of Rajasthan. Though it was reported that the find had been destroyed, the Medieval Centre had, in early 2006 acquired a small sample which, on analysis, turned out to be sulphur.

Jaisalmer and the medieval gunpowder

The Group's first visit was to Jaisamler. Attempts to make contact before leaving Europe had proved futile so the Group had to make an unannounced visit. Unfortunately the Director of the Museum in Jaisalmer Fort was not available while we were there. However the story of the reported gunpowder appeared to be well known and we talked to a number of people who claimed that they knew what had happened (figure 1). Unfortunately the stories were rather contradictory with one lurid account telling of an innocent workman using a pick to break up the gunpowder and blowing him and 2 other workmen to kingdom come. It was a salutary lesson on the speed with which oral history can operate, and the how little it can be trusted!

Figure 1. The wall of the JaisalnerFort where we were told that the 'gunpowder' had been found

Jalaser and saltpetre

From Jaisalmer the Group then met up with a Danish colleague, Torben Sode, to explore the area to the north east of Agra – from Firozabad to Purdalpur, both centres of the glass and glass bead industries. Torben introduced us to a man who supplied chemicals to the glass industry of the area and he was able to put us into contact with a company which supplied saltpetre to the glass industry in the town of Jaleser, some 25km to the north of Firozabad.

On arriving in Jalasar we were taken to a small, somewhat primitive factory where we were told that saltpetre was made and this turned out to be true. However, disappointingly the starting point was the addition of nitric acid to potassium chloride:

$$HNO_3 + KCl \qquad KNO_3 + HCl$$

The process is done at high temperature – around 60°C. Once complete the liquid was siphoned off into large plastic drums immersed in a cold bath kept at minus 10°C – maintained by an antiquated ammonia pressure system! The saltpetre precipitates out of solution and the resultant crystals, which are about 90-95% pure, are collected and bagged.

From here we were then taken to a second factory where saltpetre is made in a 2-stage process. First calcium carbonate is added to nitric acid to make calcium carbonate:

$$2HNO_3 + Ca(CO_3)_2 \qquad Ca(NO_3)_2 + H_2O + 2CO_2$$

The resultant liquid is then added to potassium chloride and heated to form potassium nitrate and calcium chloride:

$$Ca(NO_3)_2 + 2KCl \qquad 2KNO_3 + CaCl_2$$

This is done at high temperature, the tank heated by the burning of dried animal dung, and the liquid siphoned off and the saltpetre crystallised out (figures 2 and 3). The reason for this more elaborate process was that the resultant calcium chloride was also extracted and could be sold to paint manufacturers generating further income. Though the starting materials were modern chemicals the process of heating with dung and the crystallisation process were interesting and similar to older techniques.

Figure 2. The second factory – heating the tank of liquid. Lars Barfod is taking a picture

Figure 3. The resultant saltpetre

However it was at the third factory that we began to have some insights into older and more primitive methods. This third factory took the impure product from the first factory and purified it to between 99 and 99.5% purity. This was done in a 2-stage process which mirrored the descriptions we have from the 17th century in Europe. First the impure saltpetre is dissolved in large steel tanks, roughly 200cm by 180cm and 50cm deep and heated – again by the use of dried dung. The temperature was first raised to about 90°C and then reduced to around 60°C. This was done, we were told, as it helps the process of settling the impurities and getting them to fall to the bottom of the tank (figure 4). No means of measuring the temperature were visible and enquiries led to the assertion that they judged temperature by experience rather than by measurement. The tank was then kept at 60°C until the specific gravity reached 1320. Although a hydrometer was available to measure the specific gravity we were told that it was rarely used and that experience was used to judge the right time.

Figure 4. The tank at the third factory containing the crude saltpetre from the first factory

When ready the liquid was siphoned off into similar sized steel tanks to cool. A lattice framework of split bamboo at 22 -30 cm centres was suspended from two poles so that it was semi-submerged in the liquid. This provided the saltpetre with suitable centres to precipitate out and we were told that this particular size of lattice was the ideal spacing (figure 5). On crystallisation the saltpetre formed very large crystals that in overall form look like icicles, a feature noted in a 16th century report in which the crystals are described as 'frozen ykles' (A.R.Williams 1975 'The Production of Saltpetre in the Middle Ages'. *Ambix*, 22: 125) (figure 6)

Figure 5. The settling tank with bamboo lattice on top

Figure 6. The saltpetre crystals

Once the saltpetre had crystallised the liquid was again siphoned off and the saltpetre crystals removed. It was then further purified by simple washing with water. This was done on a special 'filter' called a chali or a bamboo chali. It consists of a series of split bamboos, approximately 50mm in diameter, laid closely side by side and supported on bricks. Beneath this the ground was arranged to form a drainage channel. Over the bamboo were 1 or 2 layers of coarse cloth - jute was used. The refined saltpetre was piled onto the chali and washed by simply pouring water through it from watering cans (figure 7). At first the wash water was re-used water from past washings but this was then changed to fresh water. The resultant saltpetre, called double refined, was very white and bright and said to be over 99% pure. The process started with 24 sacks of the impure saltpetre and they obtained 20 sacks at the end, the remainder was still in the various wash waters. However all the wash waters were constantly re-used in different stages of the process and over a period of about a month most of this saltpetre was re-claimed so that wastage was quite low – in the region of half a sack. No weights are used but each sack probably contained in the region of 50kg.

The saltpetre was then dried by a succession of three different methods. First it was put in a centrifuge, followed by drying in the sun and finished off by electric heaters (figure 8). Finally the saltpetre was ground up and bagged for sale.

Figure 7. The bamboo chali

Figure 8. Drying the saltpetre in the sun

The actual refining process, though it started with modern chemicals, is very close to that described in early reports. What was surprising though was that there was an unused large iron pan supported on a brick structure which looked exactly like a photograph of saltpetre making from the early 20th century (Marshall 1917 – see Appendix 1) (figure 9). This surprising find led us to hope that perhaps we could find more remains of the older, more primitive methods of extraction and refining of saltpetre.

Figure 9. The old iron pan at the second factory

Finally after further enquiries we were taken to another part of Jalesar and shown a complete saltpetre making 'factory' in which saltpetre was both extracted and refined in the traditional way. Much to our surprise we were told that the site was still working and producing saltpetre from the soil of the area.

Extracting saltpetre from the soil

The saltpetre was extracted by a process of lixiviation. This was carried out in a rectangular area some 225cm by 180cm, though we were told that this was not fixed and some were larger and some smaller, surrounded by a low dried-mud wall some 30cm high. The floor slopes slightly from the rear to the front and there is a drain through the centre of the lower wall. Over the floor of this chamber was laid a layer of thin wood branches, 1-2cms in diameter, laid across the rectangular area and supported on rows of bricks (figure 10). Over the branches was laid a layer of cloth - the same as in the chali as described above (figure 11). The soil to be extracted was then laid in a layer approximately 20cm thick over the chali and carefully compacted and smoothed by hand (figures 12 and 13). A piece of the same cloth was then laid over the soil and water poured over it – the cloth ensuring that the water soaked through the soil layer slowly and evenly without causing disturbance to the surface of the soil (figure 14).

Figure10. Laying a layer of sticks over the bricks

Figure 11. Sacking is laid over the sticks

Figure 12. The saltpetre soil is put over the sacking

Figure 13. The saltpetre is carefully packed down

As the water percolated through the soil it drained into a pot set in a pit in front of the extraction pit (figure 15). In the example we were shown the base was lined with a plastic sheet which also lined the base of the drain. Formerly the floor was just beaten and dried mud and the drain was lined with large thick leaves pegged in place with small twigs.

Figure 14. Pouring water over the saltpetre soil

Figure 15. The extracted saltpetre liquor a being collected

Precipitating the saltpetre

Once collected the liquor was transferred to an iron pan, in this case approximately 1m in diameter, and the liquid was boiled down till, by experience, it had become a saturated solution (figures 16 and 17). The pan was then taken from the fire and left to cool overnight. The water in the pan was then poured off into another container and the precipitate tipped out onto the ground (figure 18). This raw, impure saltpetre, called *jharia*, still contained a great deal of impurities and it was sold in this form to the refiners.

Figure 16. The pan for produing the crude saltpetre

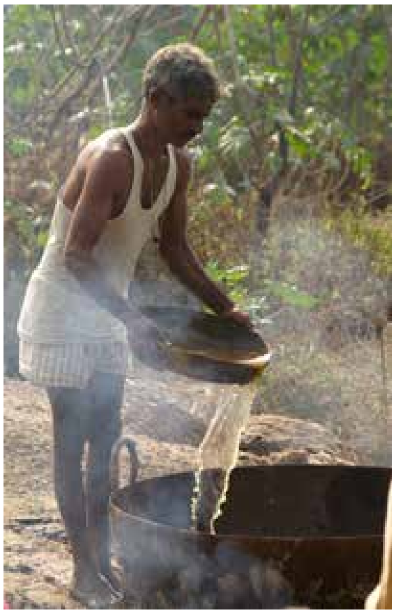

Figure 17. Pouring the saltpetre liquor into the pan

Figure 18 The crude saltpetre – the *jharia*

Refining the raw saltpetre
To refine the *jharia* it was dissolved in water in a large iron pan, some 440cms in diameter and 65cm deep, supported on a circular dried mud wall and covered with a simple thatch roof (figure 19). Beneath the pan a fire was lit and the solution heated. Both wood and dried animal dung were used as fuel. Beneath the fire the ash was collected in an ash pit

Figure 19. The iron pan used to purify the saltpetre under its thatched roof

The solution was heated for several hours. As the concentration of the solution increased the less soluble salts, particularly salt, sodium chloride, precipitate out and fall to the bottom. These impurities are scooped out periodically and heaped into baskets set on the side of the pan supported on long wooden poles (figure 20).

Figure 20. Removing the salt and impurities with a large scoop

The specific gravity of the solution is estimated by dipping a finger into it (figure 21). If the liquid dripped then it was not ready, but when the liquid stayed on the finger and congealed then the liquor was transferred by scoop, to a large wooden settling tank set behind the pan (figure 22).

Figure 21 Testing the solution

Figure 22. Scooping out the liquid into the settling tank

The solution was stirred several times in the tank and was left there to settle for about 3 hours to allow large impurities to settle out (figure 23). It was then drained into a smaller wooden tank by means of a rotating iron pipe which was turned so that just the surface of the liquid was drained. Using buckets the liquid was then transferred to a large wooden tank, the precipitation tank, set in the ground (figure 24). A split bamboo frame was suspended in the top surface of the liquid and the solution left to settle for up to 8 days, depending on the season, taking a shorter period in the winter and longer in the summer (figure 25).

Once the saltpetre had crystallised it was transferred to a bamboo chali where it was washed in cold water to remove further impurities as described above (figure 26). Samples of the materials produced by each of the processes were collected and will be analysed on our return to Denmark. Of particular importance is the level of purity of the final saltpetre and just what contaminants it contains.

Figure 23. The settling tank

Figure 24. Collecting the liquor from the settling tank

Figure 25. A precipitation tank with a lattice of bamboo on the surface of the liquid

Figure 26. The bamboo chali where the saltpetre is purified still further

Where does the saltpetre come from?

Of particular interest to us was where the actual saltpetre derives from. With the assistance of Professor Balasubramanium we were able to learn that the saltpetre soil was collected in the months of March and April. This was somewhat surprising as the sources indicate that it was collected at the end of the rainy season which finishes in September. We further learned that the process of collecting the soil and carrying out the extraction and first, crude, purification was carried out by a group of itinerant workers, from Bihar, who arrived each year in March and April. The area where they collect the saltpetre varies from year to year and we were taken to, and collected samples from, an area that had been exploited in 2004 (figure 27).

Figure 27. Taking samples of the soil for analysis

A possible reason why the soil is collected in March and April may relate to the process by which the saltpetre comes to the surface. From June to September much of India is subjected to the monsoon rains in which the ground becomes highly saturated. The water will penetrate deep into the ground dissolving salts as it does so. When the rains stop the ground starts to dry out and as the water evaporates from the surface, it will draw, by capillary action, the water deep in the soil to the surface, carrying the soluble salts with it. This drying process would seem to take several months with the most salts, including the saltpetre, collecting at the surface when the ground is driest – in March and April – and before the next rainy season.

What was also surprising was that no source of potassium, for example potash, was added during the process but that the end result was potassium nitrate - we would expect calcium salts to predominate. This question was discussed with Professor Rajiv Singha of IIT-Kanpur who believes that the answer may be that the area is fed by rivers that run through areas rich in potassium salts. But this does not answer the question of where the nitrate comes from. It is not a naturally occurring salt and is produced by the reaction of bacteria with area and ammonia compounds in animal waste, particularly urine. Was the source of the nitrate here also from this process or were there other factors at work? A possible explanation is that the very high population, of both people and animals, has over a very long period, possibly millennia, produced a high level of nitrate in the soil and this is still coming to the surface.

This information has led us to further questions and speculation about the source of the saltpetre and to the collection of more samples. In particular a series of some 10 samples of soil were taken at 1km intervals along the road from Jalesar to Purdalpur – an area from which, we were told, saltpetre soil was regularly collected. Accurate GPS positions were taken for each sample. Our intention is that a second set of samples, taken in March/April, may help to answer the question of the concentration of saltpetre at the soil surface.

Serampur

The Danish colony of Serampur, to the north of present day Kolkata (Calcutta), had been established in the 18th century as a base for trade between Denmark and the East, in particular for the exploitation of the saltpetre trade across north eastern India. However the British has established themselves in Fort William (the site of present day Kolkata) and within a short time had a strangle hold over the north east of India from which they were able to control much of the trade making Serampur unprofitable and unsustainable. It was therefore sold to the British. The primary objective of the first Galathea expedition was to officially hand over the Colony and take on board those of the Danish colony who wished to return to Denmark. In carrying out the task, the ship made a circumnavigation and conducted research, took observations and collected samples in its travels.

We managed to travel to Serampur and see what remains of the Danish colony – notably the College set up by Frederick VI in 1827, the fourth Danish university to be founded and the first European university in India, and the church built at the beginning of the 19^{th} century by Olaf Bie (figures 28 and 29).

Figure 28. The College at Serampore

Figure 29 The Church built by Olaf Bie

Conclusions

Our expedition to India was highly successful. We were able to fully document the craft of saltpetre making and record processes that have remained largely unchanged for several hundred years and which will probably die out in the near future as modern methods take over. Our study has also given us enormous insights into the way that saltpetre was extracted and purified in the early modern period and we will be able to use them in our work on medieval gunpowder from Europe.

Just as important, now that the processes are clear, we will be able to make both qualitative and semi-quantitative estimates of the industry and its production leading to greater understanding of just how India was able to produce the huge amounts of saltpetre that Europe required. This saltpetre, which was used to make gunpowder in Europe, enabled them, not only to fight their wars at home, but to carve out huge global empires whose boundaries and subsequent break up, shape not just the physical but, crucially the political makeup of much of the World today.

The future

As with most work of this nature, our visit threw up as many, if not more, questions, than it answered. The actual source of the saltpetre is especially problematic and we hope that the analysis of the soil samples we collected may go some way to answering it. Taking further samples in the same places in March/April, with the help of the GPS positions, may also help to answer the question of the mechanism of how saltpetre concentrates on the surface. Equally important will be to return to India in the March/April period when we will be able to see the process in full production. Some of the sample analysis will be carried out at IIT-Kanpur under the supervision of Professor Balasubramanium and this collaboration will continue in the future. Especially important will be the location of other places where saltpetre is still being made in the traditional manner – our visit has already resulted in a contact who claims that it is still being done in the Patna area.

All the work undertaken was fully documented and a DVD of the expedition produced.

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Appendix 1

The following illustrations are taken from the book, *Explosives* by Arthur Marshall, published in 1917. They show the traditional methods of the extraction of the saltpetre from the soil and the purification of the extracted saltpetre.