Medieval Gunpowder Research Group

Sulphur refining

Report Number 13 - September 2015

Middelaldercentret, Nykøbing, Denmark

Introduction

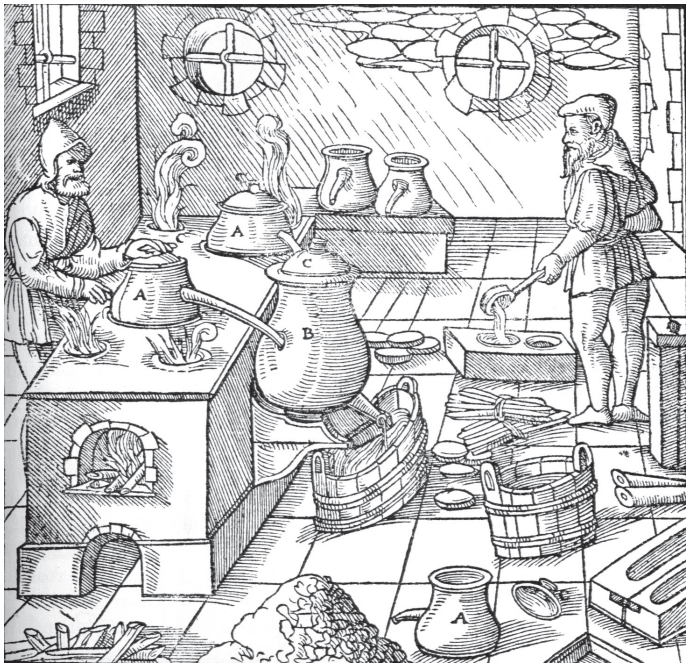
As part of our continuing work on the history and development of medieval gunpowder we have turned out attention to the production of sulphur. We had looked at this aspect of the subject before and done some work on where it was found and how it was refined. Field trips to Sicily in to Iceland in 2006[1] and Sicily in 2007[2] resulted in work on refining and production of pure sulphur. Initially we refined the raw sulphur mineral, collected in Iceland, by a simple process of melting, removing the impurities that rose to the top and pouring the liquid sulphur through a cloth sieve. The sulphur produced this way must still have contained some impurities but was found to work perfectly well in black powder recipes. In 2006 we attempted to replicate a process illustrated and described by both Agricola and Biringuccio. Unfortunately, the vessels used were insufficiently strong to withstand the temperatures required and cracked at an early stage in the process. Experiments this year were to try to replicate the process, building on the work carried out earlier.

Sources

We have two major sources for the refining of sulphur in the early modern period - Agricola's *De Re Metallica* and the *Pirotechnia* by Biringuccio. Although there are small differences between the two, they both describe essentially the same process - heating the sulphur ore to boiling point and collecting the resultant vapour and letting it run out as pure liquid sulphur.

> The ores which consist mostly of sulphur and of earth, and rarely other minerals, are melted in big-bellied earthenware pots. The furnaces which hold two of these pots, are divided into three parts; the lowest part is a foot high, and has an opening at the front for the draught the top of this is covered with iron plates, which are perforated near the edges, and these support iron rods, upon which the firewood is placed. The middle part of the furnace is one and a half feet high, and has a mouth in front, so that the wood may be inserted; the top of this has rods, upon which the bottom of the pots stand. The upper part is about two feet high, and the pots are also two feet high and one digit thick; these have below

Raw sulphur mineral collected in Iceland



Refinng sulphur - an illustration from Agricola's *De Re Metallica*.

> their mouths a long, slender spout. In order that the mouth of the pot may be covered, an earthenware lid is made which fits into it. For every two of these pots there must be one pot of the same size and shape, and without a spout, but having three holes, two of which are below the mouth and receive the spouts of the two first pots ; the third hole is on the opposite side at the bottom, and through it the sulphur flows out. In each furnace are placed two pots with spouts, and the furnace must be covered by plates of iron smeared over with lute two digits thick; it is thus entirely closed in, but for two or three ventholes through which the mouths of the pots project. Outside of the furnace, against one side, is placed the pot without a spout, into the two holes of which the two spouts of the other pots penetrate, and this pot should be built in at both sides to keep it steady. When the sulphur ore has been placed in the pots, and these placed in the furnace, they are closely covered, and it is desirable to smear the joint over with lute, so that the sulphur will not exhale, and for the same reason the pot below is covered with a lid, which is also smeared with lute. The wood having been kindled, the ores are heated until the sulphur is exhaled, and the vapour, arising through the spout, penetrates into the lower pot and thickens into sulphur, which falls to the bottom like melted wax. It then flows out through the hole, which, as I said, is at the bottom of this pot; and the work¬man makes it into cakes, or thin sticks or thin pieces of wood are dipped in it. Then he takes the burning wood and glowing charcoal from the furnace, and when it has cooled, he opens the two pots, empties the residues, which, if the ores were composed of sulphur and earth, resemble naturally extinguished ashes; but if the ores consisted of sulphur and earth and stone, or sulphur and stone only, they resemble earth completely dried or stones well roasted. Afterward the pots are re-filled with ore, and the whole work

is repeated.

(*De Re Metallica* of Georgius Agricola, Translated by H C Hoover and L H Hoover, 1950 Dover Publications, New York, pp 578-80).

Method

Essentially the method is to heat the raw sulphur to above the boiling point of sulphur, 444°C (832°F), and to then allow it to condense in the receiving vessel. Only the sulphur in the raw material would become vapourised so that the condensate should be pure sulphur.

The vessels

Two vessels were made by master potter John Hudson (http://www.hudsonclaypotter.co.uk) based on the illustrations. We decided that we would just make one heating vessel and one collecting vessel. The former had a spout which fitted into a hole in the side of the receiving vessel which had a drain at its base. The vessels were made of earthenware with a high proportion of grog to help withstand the temperatures involved. The vessels were left unglazed except for the lower inside of the receiving vessel.

The set-up

The heating vessel was filled with 5kg of the raw sulphur ore before being set up on a fire bed, the heating vessel held in a circular iron ring. The receiving vessel was set below and the spout fitted carefully into the hole at the side. A ceramic vessel was set below the outlet on the receiving vessel. The gaps around the lids and where the spout entered the receiving vessel were sealed using a flour and water paste.

The two pots set up over a hearth - unfortunately, we could not build an enclosed hearth as shown in the illustration from *De Re Metallica*

Heating

Charcoal was used as a fuel and initial heating was kept slow to prevent thermal shock and possible cracking. After about an hour of slowly increasing the heat at the base of the pot the fire was at full heat and the sulphur was left to boil. Some 3½ hours after starting a small amount of liquid sulphur, 330g, flowed into the ceramic collecting bowl but, after about 30 seconds this flow dried up.

Heating the sulphur

The small amount of sulphur that condensed in the collecting vessel before the spout became blocked

We assumed that we had not heated the sulphur sufficiently we continued to continue heating with no further outcome. Eventually the heating vessel cracked and liquid sulphur leaked out causing a small fire which had to be extinguished.

Conclusion

Following the completion of the experiment, we looked at what we had done and why it had, largely, failed. The reason was very obvious - the raw sulphur had vapourised but had then condensed in the spout and blocked it preventing further sulphur getting through. What is clear is that the whole of the heating vessel, including the spout, must be kept above the boiling point of sulphur, 444°C (832°F). We believe that essentially the process worked and it was only the fact that we did not make sure that the spout was hot enough so that the sulphur vapour would not condense until it was in the second vessel.

1 See Ho report 5 September 2006

2 See R D Smith 2010 Rewriting the history of gunpowder. Middelaldercentret, Denmark

Medieval Gunpowder Research Group - 2015

Contact list

Peter Vemming Hansen – Director mag. art.
Middelaldercentret, Ved Hamborgskoven 2, DK 4800
Nykøbing F, Denmark
Telephone +45 54 86 19 34
peter@middelaldercentret.dk

Kay Smith – Independent scholar
Hawthorne Cottage, Moorfield Road, Leeds, LS12 3SE, UK
Telephone +44 (0)113 263 7547
smithbrown@basiliscoe.fsnet.co.uk

Axel Müller – Congress Director
Institute for Medieval Studies, University of Leeds, Leeds, LS2 9JT
Telephone +44 (0)113 343 3614
a.muller@leeds.ac.uk

Ruth Brown - Independent scholar
Hawthorne Cottage, Moorfield Road, Leeds, LS12 3SE, UK
Telephone +44 (0)113 263 7547
smithbrown@basiliscoe.fsnet.co.uk

Jens Christiansen - Blacksmith
Middelaldercentret, Ved Hamborgskoven 2, DK 4800
Nykøbing F, Denmark