Chemistry in Ancient Times: The Development of Blue and Purple Pigments

Heinz Berke*

Dedicated to Professor Roald Hoffmann on the occasion of his 65th birthday

1. The World of Colors and the Antique Blue and Purple Pigments

Colors are fascinating. At first man only saw the quality and variety of colors in nature, but later tried to make them himself. That is why a special relationship exists between man and colors, both through perception and through expression.[1] Both are linked to aesthetics and have influenced the arts in various ways throughout the history of mankind. As a result, colored substances and pigments were highly valued and much sought after in ancient times as raw materials for the arts.

In prehistoric times and in the antiquity, the availability of these substances was the key problem, directly linked to their accessibility. Earth colors were readily available at all times as they could be directly taken from the soil. They were, for example, used in cave paintings. Looking at these paintings, it is striking that no blue color is found. In general, organic or mineral sources for stable blue dyes and pigments are exceptionally rare.

In ancient times, the rare blue mineral pigments could be mined to the major part only from deposits that were difficult to access. Even later civilizations often suffered from shortages of stable blue pigments. This situation did not change until the 19th century when industrialization led to the chemical mass production of dyes and pigments. The shortage of blue pigments most probably caused their high idealistic and materialistic esteem. The first blue mineral pigment that was both accessible to mankind and truly stable, was most likely lapis lazuli ($(Na,Ca)_8[SiAlO_4]_6(S,SO_4)$; S_3^-- or S_2^--chromophores embedded into sodalithe cages).[2] This precious stone was mined in the antiquity in the area of today's Afghanistan. The demand for blue pigments was also met by using the more abundant mineral azurite, a basic copper carbonate. The less stable azurite is found, for example, as a component in layers of paint of Chinese art objects from pre-Christian times. In these times, one was also able to prepare blue-colored glazes and glasses by the use of cobalt minerals. The blue glaze of the tiles of Babylon's Iŝtar gate contain significant amounts of cobalt.[3] To a certain extent smalt, a cobalt–aluminum spinel compound, was used in ancient Egypt.[4]

The blue pigments used in pre-industrial times all suffered from specific restrictions that hindered their broader application. The lack of a blue pigment that could be used universally, apparently resulted in the development of methods to produce stable blue materials. These methods also include the production of Maya Blue[5] used by Indian civilizations, which is based on indigo stabilized by intercalation into clays.

People's efforts to enhance the availability and quality of blue pigments by producing them themselves began in predynastic ancient Egypt more than 5200 years ago.[6] At that time a pigment was created which is known today as Egyptian Blue ($CaCuSi_4O_{10}$).[7–13] Traces of the compound have been found in artifacts such as a small plate from 3600 BC for an olive oil container, which certifies the quality of the oil blessed by the goddess Iset. Egyptian civilizations used Egyptian Blue frequently over the next millennia. The golden age of its use was most likely the period of the New Kingdom (1580–1085 BC), which coincides with the most productive period of artwork in ancient Egypt. Among the most remarkable pieces from this time are the crown of Nefertete and the Talatat stones of the temple of her husband Echnaton, both painted using Egyptian Blue.[6, 14]

Even before the collapse of the Egyptian Empire Egyptian Blue reached the ancient Greek and Roman civilizations.[9] Egyptian Blue was also widely used in Mesopotamia and in the area of today's Iran. Concurrently it became a commodity for the Romans. In his legendary work "De Architectura" from 24 AD, the Roman architect Vitruvius published a recipe for Egyptian Blue, known to the Romans as *caeruleus*, and also mentioned its production in factories. Together with the end of the Roman Empire, the knowledge of Egyptian Blue was lost.

Blue pigments also played an important role in China's historical development. While the already mentioned azurite was used in ancient times as mineral blue pigment, cobalt oxide found use in glazes and glasses. Azurite, relatively abundant China, was mined mainly to produce copper and copper alloys.[15] However there was no mineral blue for universal use in ancient China as azurite is rather unstable and

[*] Prof. Dr. H. Berke
Anorganisch-chemisches Institut
Universität Zürich
Winterthurerstrasse 190, 8057 Zürich (Switzerland)
Fax: (+41) 1-635-6802
E-mail: hberke@aci.unizh.ch

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the staining by cobalt oxide is limited to special applications. These disadvantages apparently triggered within the Chinese civilization the start of the industrial–chemical development and production of blue pigments with enhanced properties. The answers to this challenge are Chinese Blue and Chinese Purple, also known as Han Blue and Han Purple.[16] Compared with Egyptian Blue these pigments contain the homologue element barium instead of calcium.[8, 17, 18] They are chemical compounds of the composition $BaCuSi_4O_{10}$[19] and $BaCuSi_2O_6$.[20]

The samples investigated so far came from blue and purple octagonal dye rods, most probably used as a commodity for paint production.[16, 18] Chinese Blue and Purple were also found in pigment layers from the Terracotta Army (Figure 1)[6, 14, 18, 21] or applied for the staining of glasses.[22] Some samples date back to the "Warring States" period (479–221 BC). The Blue and Purple were most commonly used during the Q'in and Han dynasties (221 BC–220 AD).[16, 21] Later use has not yet been proven.

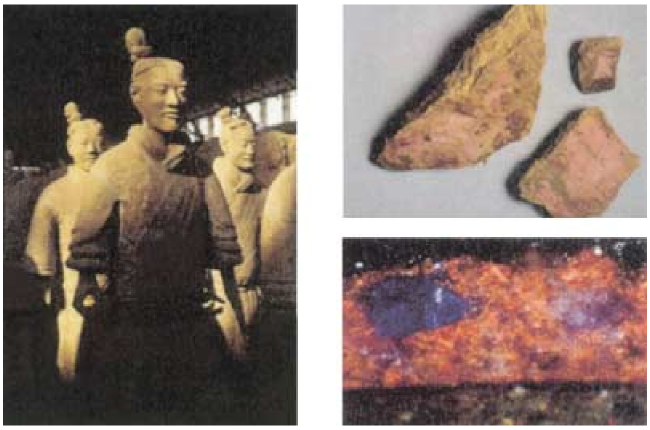

Figure 1. Sample of the Terracotta Army Xi'an, China. Left: a group of terracotta soldiers. Top right: fragments of the purple trousers of a soldier (fragment 003–92). Bottom right: A microscopic view (enlargement 500 - times) of a cross section of its pigment layer. The displayed part has a horizontal extension of 0.22 mm. The pigment layer contains grains of Chinese Purple and Cinnabar. Under the pigment layer there is a dark lacquer and under that the terracotta.

Figure 2. Egyptian Blue ($CaCuSi_4O_{10}$; left), Chinese Blue ($BaCuSi_4O_{10}$; middle), and Chinese Purple ($BaCuSi_2O_6$; right). Egyptian Blue and Chinese Blue appear very similar under comparable conditions. That they appear different here is a result of the samples being of different particle size. Small particles lead to lightening in tone. The sample of Egyptian Blue consists of coarse crystalline material, while Chinese Blue was ground.

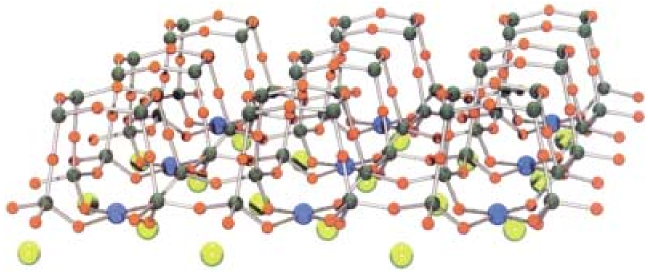

Figure 3. Schematic representation of a puckered layer of $MCuSi_4O_{10}$ (M = Ca, Ba) with Cu^{2+} ions (blue) in square-planar environment. The coordination sphere of the M^{2+} ions (yellow) is extended to eight-coordination by binding to a neighboring layer (O red, Si green).

2. Three Pigments—One Chemistry

As Egyptian Blue, Chinese Blue, and Chinese Purple are all alkaline-earth-metal copper silicates, they are chemically related compounds. Samples of the compounds are shown in Figure 2. Egyptian and Chinese Blue even have the same copper and silicate stoichiometry and the same microscopic structure. They are also very similar macroscopically, crystals of both compounds show the same appearance.[7, 17] Their structures contain four-membered $(SiO)_4$ rings and oxygen bridges linking different rings in such a way that four four-membered rings form an eight-membered ring.[6, 19] The infinite layered assembly of the rings shows puckering (Figure 3), with the remaining terminal oxygen atoms of the SiO_4 tetrahedra functioning as coordination sites for the metal ions. Between two opposing four-membered rings lies a Cu^{2+} ion in a square-planar environment. The Cu^{2+} ions occupy half of the holes and are arranged in rows. Eight-coordinate M^{2+} ions (Ca^{2+}, Ba^{2+}) occupy the remaining half of the hole sites and additionally interconnect two neighboring silicate sheets. The Cu^{2+} ions in both Egyptian and Chinese Blue have virtually identical environments. As the Cu^{2+} ions are the chromophores responsible for the blue color,[23] both compounds show very similar color properties.

Comparing compositions, Chinese Purple ($BaCuSi_2O_6$) contains two equivalents of SiO_2 less than the blue compounds. It appears during the reaction leading to Chinese Blue and is therefore a kinetic product.[6] Even though Chinese Purple also forms a layered arrangement, the structural motifs differ significantly from those of Egyptian and Chinese Blue. The condensation process to give silicate frameworks stops with the formation of islands of $Si_4O_{12}^{8-}$ rings; Cu_2 units hold these rings together to build up $Cu_2Si_4O_{12}^{4-}$ layers (Figure 4). Barium ions are located in

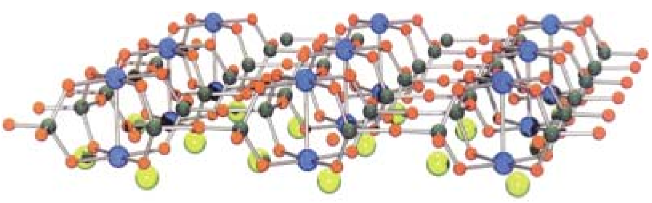

Figure 4. Layered structure of Chinese Purple $BaCuSi_2O_6$. The main feature is the Cu_2 unit (blue) which is held together by four bridging SiO_2 moieties from the four-membered silicate rings (Ba yellow, O red, Si dark green).

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between the sheets and interconnect them in a way similar to that of Chinese Blue. The unusual and unique feature of this compound is the Cu–Cu bond with a bond length of 2.73 Å, very close to the reference compound copper acetate ($Cu_2(acetate)_4$, 2.64 Å).[6, 18, 20] Thus, the early Chinese chemists—whether they deserve this title on closer inspection remains doubtful—were the first to prepare a chemical compound containing a metal–metal bond.

The instability of Chinese Purple is to a great extent attributable to the presence of the Cu–Cu bond. For example, the compound is sensitive not only to mineral acids but also to oxalic acid. This might be the reason why microorganisms that excrete oxalate or oxalic acid are suspected to contribute to the decay of works of art.[24] Chinese Purple is also thermally less stable than Chinese Blue as it decomposes at 1200 °C within 4 hours to generate a green-black glass. It is likely that Chinese Blue is formed to some extent as the reactions in Equations (1) and (2) take place.[6, 18]

$$3\,BaCuSi_2O_6 \xrightarrow{\Delta} BaCuSi_4O_{10} + 2\,BaSiO_3 + 2\,CuO \qquad (1)$$

$$2\,CuO \xrightarrow{\Delta} Cu_2O + \frac{1}{2}\,O_2 \qquad (2)$$

Chinese Blue is stable under these conditions, but Egyptian Blue decomposes at 1000 °C[7] to a green-black mixture of copper oxide, tridymite, and glass. This property sets strong restrictions to a successful syntheses of the compound because temperature control is required. It also caused in ancient times the need to develop special processing methods for the production of compact bodies of Egyptian Blue such as amulets, seals or bricks (Figure 5). As simple casting was not possible, these objects had to be made by complicated multistep sintering processes.

Figure 5. Amulet of dwarfish god Bes consisting of compact Egyptian Blue, 24th Dynasty (Property of the author).

3. *Complex Experimentation*

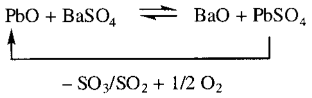
The ancient task of developing methods to produce Egyptian Blue, Chinese Blue, and Chinese Purple faced more than the general problems normally associated with chemical synthesis. In the case of Egyptian Blue the starting materials, lime, sand, and copper minerals (azurite, malachite), were easily accessible in ancient times.[7] In some places,[25] azurite is even found as a mixture together with lime and sand. Such a three-component mixture might have been used directly for the synthesis, but how the preparations were developed remains unknown. We can only speculate that this favorable natural circumstance might have promoted the early chemical approaches. From our experience in chemistry today we know that any basic discovery by chance is only a first step towards useful products. Following the first discovery of Egyptian Blue, it was necessary to work out the stoichiometry of raw materials and flux additives for the pseudo-sintering process, as well as physical and chemical conditions for the synthesis. This was surely a tedious development lasting generations. However, it is important to note that, based on our own archeometric measurements as well as those of other groups, the composition of Egyptian Blue samples is quite constant over 4000 years. This indicates that it was soon realized how important it was to use constant proportions of the raw materials. For example, the blue pigment of Nefertete's crown has a composition very close to $CaCuSi_4O_{10}$.[6] Nevertheless, some investigations have shown that ancient samples of Egyptian Blue often contain wollastonite ($CaSiO_3$), because too much lime and quartz were used.[11]

The synthesis of Egyptian Blue became more efficient by the use of a flux additive. As independent experiments demonstrate, this also significantly enhances the quality of the product. Specifically, papyrus ash (consisting mainly of K_2CO_3), NaCl, and the ancient trona (a mixture of Na_2CO_3, Na_2SO_4, and NaCl from the Wadi Atrun)[7, 9, 12] were used in ancient times. This important refinement of the production method was without doubt the result of lengthy empirical developments. Additionally, the synthesis requires a supply of air and a firing temperature between 800 and 900 °C. In the time of the Old Kingdom (3197–2778 BC) this was realized by blowing air in through pipes. Later this was replaced by bellows. Direct ways to measure the temperature (like thermometers) were unknown to ancient Egyptian civilizations so other criteria were relied upon to indicate whether the correct temperature had been reached. Since the production methods were empirical, specialized personnel was required for development and realization. As a result, a good technical "infrastructure" and a high degree of craftsmanship was necessary.

The production of Chinese Blue and Chinese Purple is even more complex than that for Egyptian Blue. One factor is that the phase diagram for $BaO/CuO/SiO_2$ shows at least four phases, so strict control of the amounts of the starting materials is necessary to obtain pure products. The two historically relevant phases that may be also found as mixtures have a Ba:Cu ratio of 1:1. It is unclear to what extent the formation of mixtures was planned to generate different color tones. The formation of mixtures may, however, happen independently of the raw-material stoichiometries, because $BaCuSi_2O_6$ is formed first as the kinetic product. $BaCuSi_4O_{10}$ is the product of a comparatively slow subsequent reaction. High temperatures (≈1000 °C) were needed to produce both pigments and this temperature had to be maintained for a significant time, in particular to obtain pure $BaCuSi_4O_{10}$.[8, 17]

clearly, the synthesis required substantial technical demands, as it still does today.

The greatest problem for the production of Chinese Blue and Chinese Purple however was the barium source. Independent experiments have shown that the use of witherite ($BaCO_3$) leads to products of satisfactory quality, but witherite is very rare and was not readily available in ancient China. Nevertheless some samples indicate its use. If baryte ($BaSO_4$) is used in a mixture with quarz and copper minerals, only small amounts of blue or purple barium–copper silicates form.[6, 14, 18] Chinese chemists nevertheless found a brilliant trick to circumvent these problems: they added lead salts (lead carbonate or lead oxide) that catalyze a dismutase reaction leading to the in situ decomposition of $BaSO_4$ (Scheme 1). Since $PbSO_4$ decomposes already at 1000 °C (much lower than the 1560 °C needed for $BaSO_4$!), it is reasonable to assume that the PbO formed is performing catalysis, while BaO is removed from the equilibrium as Chinese Blue and Chinese Purple are formed.



Scheme 1. Decomposition of $BaSO_4$ with lead catalysis.

There is still no answer to the fundamental question as to why Chinese chemists considered including barium minerals into their syntheses. It is not an absolutely impossible idea to assume that these pigments were discovered in a totally independent way by applying the rare barium minerals. This is not very convincing though if an alternative theory is taken into account that will be presented in the following section. In either case, the use of barium minerals requires mineralogical knowledge to be aware that these substances are special materials. Some general interest in barium minerals related to glass production is recorded in China since the Warring States period.[22, 26]

4. Technology Transfer

It was already mentioned that the use of Egyptian Blue had spread all over the Mediterranean area by the first millenium BC and was employed by many civilizations. Egyptian Blue of high quality was obtained at this time and as long as no compact bodies had to be produced, the physical and chemical conditions for the synthesis were relatively easy to attain. Additionally, the raw materials were easily available. The situation was fundamentally different for Chinese Blue and Chinese Purple at the beginning of the Warring States period. The syntheses of these color pigments were much harder concerning restrictions imposed by restraints from the stoichiometry, the more difficult physical conditions, and the availability of the raw materials. This explains why it seems quite unlikely that the invention of these pigments was accomplished in a convergent manner more or less ab initio. Knowledge about the many conditions for the syntheses, which includes the restrictions arising from the available raw materials, had to merge together at the same time in one experiment to successfully generate the pigments.

On this basis, it seems more likely to assume that the production methods for Chinese Blue and Chinese Purple were developed stepwise and that they were based on the methods to produce Egyptian Blue. From today's perspective, the blue compounds only differ because of the chemically minor variation of the alkaline-earth metal. Considering the knowledge of the times, the modification of Egyptian Blue has to be viewed as a big step as there was no knowledge of atoms and molecules or even the periodic table. Therefore, a very tedious empirical approach was the only alternative.

Further facts support the hypothesis about the relationship between Egyptian Blue and the Chinese pigments. Two antique objects made from Egyptian Blue, a cup and a sistrum from Hasanlu in Iran,[12, 27] both first of all demonstrate the use of this synthetic pigment in regions far to the East of Egypt. Additionally, these objects show a chemical variation of Egyptian Blue ($CaCuSi_4O_{10}$), since they at least to some extent contain the strontium analogue $SrCuSi_4O_{10}$.[28] It seems quite reasonable that the replacement of calcium by strontium was not planned but instead happened by chance. But still it happened and most probably based on the knowledge about Egyptian Blue.

The question remains as to how knowledge about Egyptian Blue spread. It might have happened along the Silk Road which was the only existing link between the Mediterranean and the Far-East. Historians suppose that the Silk Road, that also passes through Iran, was used from at least 1000 BC. This is much earlier than the occurrence of Chinese Blue and Chinese Purple during the Warring States period. In principle, other similar transfer events give further support to this idea of a technology transfer along the Silk Road. However, in such other cases the information was transferred in the other direction from the Far-East to the Mediterranean. Examples include the transfer of knowledge about silk[29] and paper[30] production.

As was already shown, some difficulties with the barium–copper silicate chemistry could not be solved on the basis of Egyptian knowledge alone. Additionally, independent developments were required. The invention of Chinese Blue and Chinese Purple is therefore an admirable technical–chemical feat. The pigment syntheses are excellent examples of the positive influence of science and technology on society, in this case related to chemistry and accompanied by technology transfer. Focusing on ancient Egypt, this occurrence is a well known fact.[31] Constantly striving for improvement is generally seen in the ancient Chinese civilization, as well.[32]

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