ADVANCES IN 13th CENTURY GLASS MANUFACTURING AND THEIR EFFECT ON CHEMICAL PROGRESS

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Introduction

The technology of glass production is thought to have been known since ~2500 BC (1-3) and had become fairly advanced within the Roman Empire (4). At that time glass was widely used for blown vessels, pitchers, beakers, bowls, and other tableware, with such glass objects becoming as widespread as pottery (1-3,5). The use of glass for laboratory apparatus, however, was rather limited because of a lack of durability under rapid temperature changes and poor chemical resistance. For example, the combination of poor quality and the thick, irregular nature of the glass resulted in the frequent breaking of the vessels during distillation (6).

With the collapse of the Roman Empire, glass production declined for a time as glassmakers moved either into the East or to the outer regions of the old empire (1, 3). These glassmakers, however, not only preserved many of the Roman glassmaking techniques, but also began developing new patterns and styles. One such glassmaking center flourished in Venice and Murano (Fig. 1) during the 13th through 16th centuries (1, 3, 7), and it was here that improved glass was produced beginning in the second half of the 13th century (3, 4, 8). The strength of the Venetian glass made it especially practical for glass vessels, and its high melting point made it useful for laboratory apparatus (1). The glass industry received great impetus from the growing general use of glass for chemical vessels. At the same time, the flourishing industry at Venice and Murano greatly influenced chemical progress (5, 7). The ability to produce more laboratory apparatus and vessels from glass allowed much greater freedom and versatility in their design; no matter what shape was needed, it could be made of glass. Additionally, pieces of glass could be melted together, forming a seal without cement. It is difficult to imagine modern chemistry without glass apparatus.

The goal of this study is to attempt to bring together various partial works in history, chemistry, and glass studies in order to give for the first time a detailed picture of how and why the Venetian glass of the 13th century

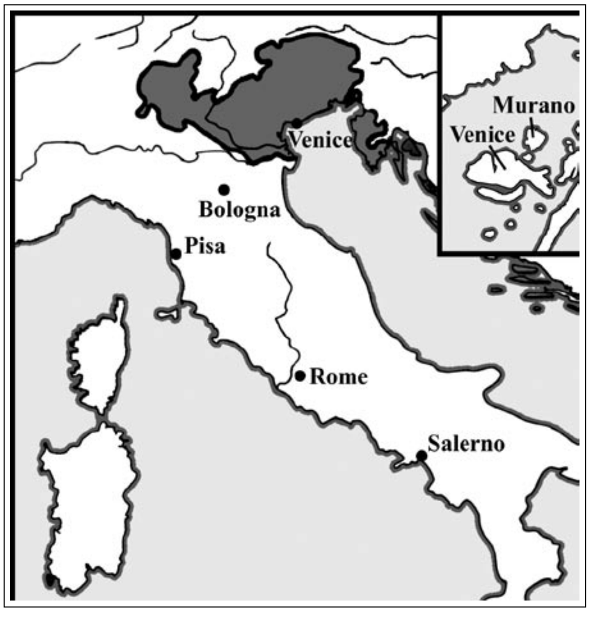

Figure 1. *The Venetian territory of the 14th century (9)*

became suitable for use in chemical apparatus and what effect this new glass had on the progress of laboratory practitioners. For example, it could be argued that this improved glass technology led to the invention of eyeglasses and a vast improvement in still design, both of which occurred shortly after the introduction of the improved glass and within close geographical proximity to Venice. As a result of better stills, important materials were isolated in pure forms for the first time, most notably alcohol and the mineral acids. The availability of these materials then greatly changed the evolving fields of both chemistry and medicine.

The Composition of Medieval Glass

The majority of medieval glasses adhered rather closely to a formula consisting of three primary components: silica, lime, and an alkali, typically either potash (K_2CO_3) or soda (Na_2CO_3) (10-12). The alkali is used to reduce the rather high melting point of the silica (~1710 °C) to below 1000^o C (10). The addition of calcium salts can result in an even greater reduction than alkali alone, resulting in the lowest temperature of the triple eutectic at ~725° C and giving a typical soda-lime glass of the composition 21.3% Na_2O, 5.2% CaO, and 73.5% SiO_2 (10).

The importance of lime, however, was not initially recognized and it was not intentionally added as a major constituent before the end of the 17th century (2, 11-13). Prior to that time, all lime content in the medieval glasses was a result of impurities in either the silica or alkali source. Until the beginning of the 14th century, the nearly exclusive source of silica used by the Venetian glassmakers was various Sicilian sands (13). These sands are thought to have provided also considerable alumina, as well as iron oxide, lime, magnesia, and frequently small amounts of manganese (2, 8). These sands were gradually replaced with quartz pebbles (~98% silica), which reduced impurities that contributed to coloring of the glass (iron, chromium, etc.) (13-15). The two primary sources of alkali were *natron*, a natural sodium sesquicarbonate ($Na_2CO_3 \cdot NaHCO_3 \cdot 2H_2O$) found in Egypt and Syria, and various types of plant ash (2, 12-17). The actual composition of *natron*, however, often varied widely because of chloride and sulfate impurities (12, 14, 15). As one might imagine, the composition of plant ash could be even more complex and variable. In addition to providing sodium and potassium carbonates, plant ash often furnished sodium and potassium chlorides and sulfates, as well as calcium and magnesium salts of carbonate and phosphate (8, 12-14, 18). These calcium salts would then be converted to lime during the fusion processes. Thus, the actual composition of medieval glasses depended heavily on both the specific raw materials and how those materials were treated prior to use.

As discussed above, the source of silica could affect the resulting glass composition. It could be argued, however, that the largest difference between the glass compositions of the previous Roman period and the improved Venetian material was due to the source of alkali. Most Roman glasses were prepared with *natron* as the alkali of choice (2, 18). The Venetians, however, favored the use of plant ashes, in particular the ashes from the salt marsh plant *salsola kali*, which were imported from the Levant (modern Syria, Libya, and Egypt) (8, 12-16, 18). These Levantine ashes, called *allume catino* (19), were used almost exclusively in Murano until the end of the 1600s (16). During this time their use was even protected by the Venetian government, and the use of other plant ashes for glassmaking was expressly prohibited (13, 16). *Allume catino* had relatively high soda content (up to 30%), as well as quite large amounts of calcium and magnesium carbonates (8, 12-14, 16, 18), but the exact source of the ash could also play a factor in its composition. For example, the ash of Syria was regarded to be better than that of Egypt, as the Syrian ash was blacker in color because of its higher carbon content (14). It has been proposed that Venice's close economic ties to the Levant, including access to Syrian glass technology and craftsmen, greatly influenced Venetian glassmaking and thus resulted in a blending of the plant ash-based methods of the Levant with the previous Roman methods (3, 4, 18).

In addition to *allume catino*, the Venetian glassmakers also utilized *barilla* or Spanish ash, obtained from the burning of marine plants (*salsola sativa, halogeton sativus, salsola kali,* and *suaeda maritima*) from the salt marshes of Alicante, Spain and other parts of the Mediterranean (14, 17). The highest quality *barilla* for glassmaking was called *agua azul*, of which Alicante was the sole source (17). The source of this form of *barilla* was described as a shrub with blue green berries, thought to be *salsola sativa,* which gave this particular *barilla* ash a blue color (14, 17). Like *allume catino*, *barilla* was a soda-rich ash (up to 30%) containing significant quantities of calcium salts (12, 15). While it is believed that both the Levantine and some *barilla* ashes may have been derived from *salsola kali*, it is important to note that plant ash composition depended largely on the soil in which the plants grew. This is best illustrated by the fact that plants grown in salty soil or near the sea produced ash high in soda, while those grown inland gave ash with higher potash content (2). As a result, the Levantine and *barilla* ashes may have been similar, but distinct, raw materials, with *allume catino* being the initial and preferred material and *barilla* becoming more common by the 16th century (12). Neri later wrote of both materials, also expressing a preference for the Levantine ash over *barilla* (20). This preference was due to the fact that glass from *barilla* would tend to suffer from some light blue coloring (14, 17, 20), which has been proposed to be a result of iron oxide content in the ash (14).

Another significant contribution to the advancement of the Venetian methods was the introduction of new processes for the preparation of the alkali raw materials. The plant ash (either *barilla* or *allume catino*) was shipped to Venice as hard pieces of calcined residue. Although these chunks of calcined residue then required pulverization after arrival, it was preferred over ash that arrived in powdered form (8, 13, 16). The pulverized ash was then purified by a series of sieving, filtering, and/or recrystallization steps, which could remove unwanted impurities and result in an alkali source with a more consistent composition.

The Effect of Composition on Physical Properties

While the typical soda-lime glass composition of 21.3% Na_2O, 5.2% CaO, and 73.5% SiO_2 can give a low melting material that is easy to work with, it does not have sufficient chemical durability to be practical. Additionally, laboratory glassware must often withstand severe temperature changes in the presence of strong reagents. Hence, for laboratory glassware to be useful, it must not only be resistant to chemical attack, but must also be durable under thermal stress.

The low chemical durability of typical soda-lime glasses is largely due to the high sodium oxide content (10, 21). Decreasing soda and increasing lime content can overcome this problem, but this defeats the purpose by increasing the tendency toward devitrification (i.e. glass crystallization resulting in frosting and loss of transparency). This, however, can be corrected by the addition of further oxides such as magnesium oxide (10).

It has been shown that replacing sodium content with either lime or magnesium results in increased resistance to attack by acidic or basic solutions (21, 22). In fact, lime or magnesium oxide content as low as 3% results in significant increases in chemical resistance. Additional magnesium content shows a slight advantage over lime for improved water and acid resistance, but lime imparts a markedly improved resistance to alkaline solutions in comparison to magnesium.

The second critical property for laboratory glassware, its thermal durability, is also dependent on chemical composition. Like most solids, glass undergoes thermal expansion that can result in increased stress during rapid temperature changes. For simple soda glasses, the thermal expansion actually increases with soda content, thus resulting in glasses with low thermal durability and a tendency to break under rapid heating. Substitution of sodium oxide by another oxide results in decreased thermal expansion with increased content of the new oxide. Both magnesium and calcium oxides, discussed above, result in significantly decreased thermal expansion, with the addition of magnesium oxide providing the greatest effect (23).

The preparation and purification methods employed by the Venetian glassmakers ensured raw materials with a more consistent composition, resulting in the production of a more consistent and uniform glass. However, the use of the Levantine and *barilla* ashes over *natron* may have played as great a role in the improvements in glass. As shown in the Table, Venetian glass samples dated to the 11th-14th century (samples B-D) exhibit considerably higher calcium and magnesium content by comparison to earlier Italian glass of the 9th-10th century (sample A) (2, 13). While the dating of samples B-D is somewhat broad, the time span does encompass the period believed to have resulted in the marked improvement in glass technology (i.e. the later 13th century) (3, 4, 8), and the changes in the composition of these later samples are consistent with the discussed improvements. In addition, it is reasonable to argue that the higher content of these elements is due to the use of the Levantine or *barilla* ashes that are known to contain significant amounts of calcium and/or magnesium (8, 12-15, 18). The higher content of these oxides would therefore result in a material that exhibited both higher chemical durability and less thermal expansion (10, 21-23). This new glass would therefore be more resistant to the action of water, acids, and bases, and would be less affected by rapid temperature changes, thus making it ideal for use in laboratory glassware. The introduction of this improved glass then paved the way for new and improved applications of its use. It has been argued that the development of both lenses and laboratory glassware was a result of these Venetian advancements (24).

Table. Chemical composition of Italian glass samples from the 9th-10th and 11th-14th centuries

Content	**Glass Sample**[a]			
(wt %)	**A**	**B**	**C**	**D**
SiO_2	77.8	68.5	68.6	70.0
Al_2O_3	2.2	1.95	1.40	1.90
Na_2O	6.4	12.5	12.5	11.7
K_2O	8.7	3.00	2.90	1.45
CaO	2.1	8.20	9.05	11.9
MgO	0.7	2.70	3.05	1.15
Fe_2O_3	0.8	0.47	0.38	0.30

[a] A: 9th-10th century (Ref. 2); B-D: 11th-14th century (Ref. 13)

Spectacles

While the specific inventor of eyeglasses is unknown, available evidence points towards their development shortly after 1286, most likely in Pisa, and their use spread rapidly throughout Europe (24-29). The production of eyeglasses was facilitated by the glassmakers' mastery of the making of uniform, clear glass necessary for a good supply of quality lenses, and it has been speculated that the original inventor was most likely an experienced glass worker (25-27). By 1300, eyeglasses were being produced by Venetian glassmakers and were repeatedly referenced in guild regulations during the first two decades of the 14th century (25-27). In fact, Venice became such an important center for the production of eyeglasses that Venetian spectacle makers left the existing glassmakers' guild to form their own guild in 1320 (26). While glassworks and the production of eyeglasses became established in other regions, the glass of Murano was considered to be of superior quality and a more suitable substance for the grinding of quality lenses. Therefore, Murano glass continued to be imported into these regions even after independent glassworks had been established (26).

The earliest spectacles were comprised of two separate lenses and frames, held together with a rivet (26, 29). These spectacles utilized convex lenses (24, 27, 28), thus improving vision for the farsighted and were used primarily for reading (26, 29). Concave lenses, for the nearsighted, were more difficult to work and did not arrive until the mid-15th century (24, 27-29). Without eyeglasses, people born with poor vision would be illiterate or have insufficient vision for a skilled trade. Even most people with normal vision typically lose the ability to focus after the age of 40 (24). Thus, eyeglasses, which nearly double the intellectual life span of the average person, affected the progress not only of chemistry, but science and technology in general.

Stills and Alcohol

The ability to use glass in the production of laboratory apparatus allowed much greater freedom and versatility in design, and nowhere was this more evident than in the rapid evolution of the still. The still, thought to have been the earliest specifically chemical instrument, dates back to the end of the first century (30-32). As shown in Fig. 2, the traditional form of the still consisted of three components: the distillation vessel (*cucurbit*), the still head (*ambix*) with an attached delivery tube (*solen*), and the receiving vessel (*bikos*). The term *ambix* was later transformed through the addition of the Arabic article (*al-*) to become *alembic,* and by the Middle Ages the term *alembic* was used to refer to the still as a whole (6, 30-32).

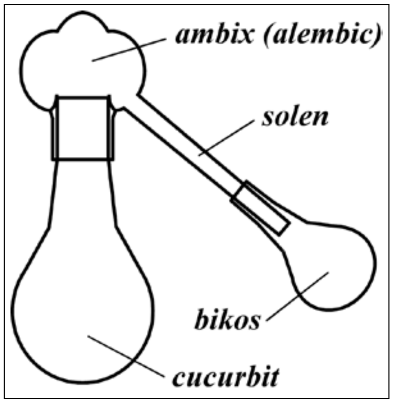

Figure 2. *Various components of the early still.*

The early stills were made from a mixture of primarily earthenware (with the interior glazed) and copper, although sometimes glass receiving vessels were used (6, 30, 31). As glass industries evolved, it became more common to use glass for first the *alembic* and then later for both the *cucurbit* and *alembic* (6). One of the difficulties encountered with the use of glass in still components was the breaking of the vessels because the glass was typically thick, irregular, and of poor quality. To counter this, a thick coating (up to two or three fingers) of clay was applied to the exterior of the *cucurbit* (6). This helped reduce breaking, but the poor heat transmission of the coating resulted in unnecessarily long preheating periods, thus making it difficult to distill volatile liquids such as alcohol.

Evidence clearly shows that alcohol was discovered ~1100 AD, most likely at the School of Salerno, the site of an important medical school (33-37). The reason for the late discovery of alcohol was partly due to the long preheating period coupled with inefficient cooling.

However, another factor was that even the most refined alcoholic distillate separated by the early stills contained so much water that it would not burn, thus making it difficult to differentiate from normal water (34-36).

Initial efforts to improve cooling methods were to cool the delivery tube (*solen*) with wet sponges or rags. As the delivery tube was now typically cooler than the *alembic*, condensation occurred primarily in the delivery tube. Because of this, the typical medieval *alembic* no longer contained an inner rim for collecting the condensate within the still head. As glass components became more common, more versatile approaches to cooling were investigated. These ideas culminated in the "wormcooler" or cooling coil, which led the cooling tube through a tub of water for more efficient cooling of the delivery tube, as shown in Fig. 3 (38). This idea was introduced in the late 13th century in the writing of Taddeo Alderotti of Florence (Thaddeus Florentinus, 1223-1303) of the University of Bologna. By use of a "canale serpentinum" run through a cooling trough and a regular supply of fresh cooling water, it is thought that it was possible for Thaddeus to obtain easily 90% alcohol after multiple fractional distillations (36, 38).

The impact of the glass industry on still evolution was evident by the move away from earthenware still components to all glass stills, which were eventually blown or cast in one piece. This new type of distilling apparatus was called the *retort* (from Latin *retortus*, "bent back") as shown in Fig. 4 (6). More importantly, later 16th-century authors such as Hieronymus Brunschwyck (1450-1512) and Conrad Gesner (1516-1565) specified not only glass distillation components, but preferably those of Venice (39, 40). Brunschwyck even stated that the distillation vessels (40):

> ..must be made of venys [Venetian] glasse bycause they shoulde the better withstande the hete of the fyre.

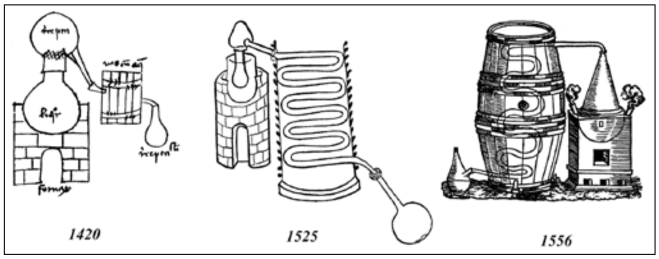

Figure 3. *15th and 16th century woodcuts illustrating the "wormcooler" cooling coil (Ref. 5, 37).*

From such writings it was clear that specialists in Venice and Murano designed and made specific glass apparatus for the practicing alchemist and artisan. The use of such apparatus then made the isolation of alcohol routine, so that it could become a common reagent of the laboratory.

The primary importance of alcohol to chemical pursuits was its use as a powerful solvent. Not only could it solubilize most salts and other water-soluble substances, but it also dissolved many organic materials not soluble in water, such as fats, resins, and essential oils. This greatly expanded the number of possible useful solutions available to the practicing alchemist and provided the first liquid known that could be used to extract the volatile aromatic substances from plants (38, 41). At the same time, alcohol began to be used as a medicine in the mid-13th century, two Italian physicians, Vitalis de Furno (ca. 1260-1327) and Thaddeus Florentinus, being the first who are known to have applied it in this way (34). It was reasoned that purified alcohol would in turn purify the patient from illness and by 1288, alcohol as a medicine was in general use. Its effect on the human organism was obvious and its effect on the failing powers of the aged led to its use as a medicine against old age. Its power of preserving organic matter from putrefaction probably also helped support the idea that it would preserve the human body. The belief that alcohol was the *quintessence* gave reason for the presumption that it would prove to be the most perfect of medicines (41). In a more practical sense, washing wounds with alcohol cleansed them and killed some microorganisms. In addition, administering alcohol to the patients made them relaxed, comfortable, perhaps even happy, thus allowing the body a chance to heal itself (37). By the mid 14th century, the medicinal and preservative properties of pure alcohol became the backbone of the writings of such authors as John of Rupescissa, and it was soon widely recommended as a universal remedy (36, 38).

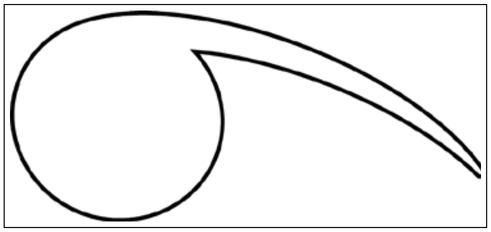

Figure 4. *The retort.*

Mineral Acids

While earlier practitioners were well acquainted with the vitriols (i.e. metal sulfates) and their calcination products, the acid vapors had not been condensed prior to the 13th century. It has been suggested that the newly formulated retort may have been important in the preparation of the mineral acids, as its one-piece design would have been beneficial for such corrosive compounds (35). Without doubt, glass or other still materials used for such isolations required good chemical resistance, which may have been the factor limiting an earlier discovery.

Nitric acid was prepared by the dry distillation of mixtures of saltpeter (KNO_3) with either alum ($NH_4Al(SO_4)_2 \cdot 12\ H_2O$) or sal ammoniac ($NH_4Cl$). The resulting acidic vapors would condense in the still head along with adventitious water, thus producing aqueous nitric acid solutions. This acid was soon produced in large quantities as a sideline of the saltpeter industry; and by the 15th century Venice had become a center for its large-scale manufacture (35, 42).

Sulfuric acid (oil of vitriol) was prepared by first "roasting" or calcining vitriol (usually green vitriol or hydrated $FeSO_4$) in an earthen vessel to produce a crude mixture of metal oxide and sulfuric acid. The mixture was then distilled in a glass retort to isolate the desired acid solution. Alternately, sulfuric acid solutions were also made by burning sulfur under a glass bell and dissolving the resulting vapors in water (35, 42).

Although the preparation of hydrochloric acid seems to have occurred at a later date and was not commonly used until the 17th century, the use of nitric and sulfuric acid reagents quickly changed the laboratory setting. Access to these acid solutions allowed practitioners to dissolve metals and most ores either at room temperature or in a water bath. This removed the need for enormous furnaces in special workshops, since glass vessels at workbenches were now sufficient for many processes. Entirely new classes of room temperature reactions were now possible, and there was an enormous increase in the number of people who could do laboratory work, thus greatly accelerating the rate of progress in chemical technology.

Conclusion

A combination of the calcium and magnesium content of the alkalis utilized and the purification of those materials to maintain consistent properties allowed the glassmakers of Venice and Murano to produce superior glasses beginning in the 13th century. The quality of the Venetian glasses dominated the European glass-making industry until the 18th century and had a direct impact on the advancement of the chemical sciences. The ability to produce more laboratory apparatus and vessels from glass allowed much greater freedom and versatility in the design of chemical laboratory ware, especially a vast improvement in stills. As a result of better stills, important materials were isolated in pure forms for the first time, most importantly alcohol and the mineral acids. The availability of these materials then greatly changed the evolving fields of both chemistry and medicine and marked the beginning of a new stage in the history of chemistry.

ACKOWLEDGMENTS

The author thanks the National Science Foundation (CHE-0132886) and North Dakota State University for financial support.

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