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Laboratory Experiment

An Experiment of Chemistry with Historical Context: 18th-Century Potash Production in Brazil

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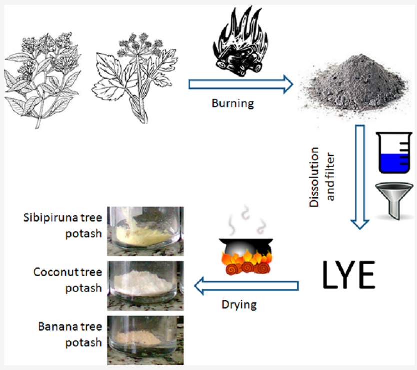
ABSTRACT: Potash, an essential raw material of the 18th century, used to be produced from the ashes of plants. Known since antiquity, it was in 1807 that Humphry Davy put an end to the decades-long controversy about its nature as a compound. The technology behind potash production was implemented in Brazil by the Portuguese naturalist Frei Velloso. The steps in potash production and the related difficulties and controversies about its composition make this case study a convenient tool for teaching the history of chemistry. The results concluded that the potash production from ashes provided proper context for teaching practical extraction yield, carbonate identification, and pH determination.



KEYWORDS: *High School/Introductory Chemistry, First-Year Undergraduate/General, Laboratory Instruction, Hands-On Learning/Manipulatives, Gravimetric Analysis, Precipitation/Solubility, Acids/Bases, Reactions*

■ INTRODUCTION

On Potash Production

Potash was arguably the most important industrial chemical product in the 18th century when it also is known as vegetable-fixed alkali. Because of its basic properties,[1−3] potash was essential for the manufacture of gunpowder and glasses, in the preparation of medicines and dyes, in the production of textiles and sugar, as well as in the bleaching of paper.

During that period, the term potash could be used to describe what today we call potassium carbonate (K_2CO_3), potassium bicarbonate ($KHCO_3$), potassium oxide (K_2O), or potassium hydroxide (KOH). The latter was called caustic potash. According to Bergman (1735−1784),[4] the word potash is a portmanteau of the words "pot" and "ash", which originated from the method used for its extraction from ashes in iron pots.

In the 18th century, the discussion was centered on whether it was a simple or a compound substance. Lavoisier, for example, did not include potash in his "Table of Simple Substances",[5] while Jane Marcet[6] acknowledged that despite the evidence indicating that potash should be a compound substance, until that moment it had not been decomposed, and should be classified as a simple substance.

As soon as it was established as a compound, different opinions about the chemical composition of potash emerged, such as those of Guyton de Morveau and co-workers,[7] who claimed that it consisted of lime and hydrogen. Humphry Davy, before he took up the experiments that would lead to the decomposition of potash by electrolysis, maintained that potash was a compound of nitrogen combined with either phosphorus or sulfur.[8]

In his landmark *Traité Elementaire de Chimie*, Lavoisier described some of the properties of potash. According to him,[9]

> *each particle of potash, at the instant of its formation, or at least of its liberation, is in contact with a particle of carbonic acid, and, as there is a considerable affinity between these two substances, they naturally combine together. Although the carbonic acid has less affinity with potash than any other acid, yet it is difficult to separate the last portions from it.*

In order to obtain the purest potash, free of carbonic acid, it was necessary to treat it with quicklime and keep it in a closed vessel. Despite having identified the strong affinity of potash for atmospheric carbon dioxide, Lavoisier ignored its composition, since[10]

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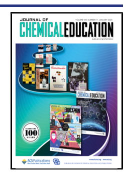

We are not better acquainted with the constituent elements of soda than with those of potash, being equally uncertain whether it previously existed ready formed in the vegetable or is a combination of elements effected by combustion.

After centuries of controversy about the nature of potash, a lasting consensus was reached when Humphry Davy established that it was possible to decompose it into its elements by electrolysis.[11]

All along the 18th century, the industrial production of potash was based on the burning of suitable plants. The ashes went through processing by leaching with water resulting in a solution called lye. The lyes thus obtained were dried to produce the impure potash, which was calcinated to finally result in pure potash. Ashes from certain plants yielded between 3% and 20% of potash, enough for most purposes. However, the process was extremely wasteful, as plant burning resulted only in a small fraction of its total weight in ashes. This method is still used nowadays in remote places to produce potash for cloth dyeing and soap making. It is noteworthy that the term "lye" can be found qualifying a solution as alkaline or naming a chemical like sodium or potassium hydroxide. This ambiguity can lead to misunderstanding. In this paper, the word lye refers to the alkaline solution produced from the aqueous extract from ashes.

All plants have some potassium in their composition, but the content varies widely. When vegetable organic matter burns, potassium oxalate ($K_2C_2O_4$) and potassium tartrate ($K_2C_4H_4O_6$) are converted to potassium oxide (K_2O),[12,13] as shown in the following simplified chemical equations (eqs 1 and 2):

$$2K_2C_2O_4(s) + O_2(g) \xrightarrow{\Delta} 2K_2O(s) + 4CO_2(g) \quad (1)$$

$$2K_2C_4H_4O_6(s) + 5O_2(g) \xrightarrow{\Delta} 2K_2O(s) + 8CO_2(g) + 4H_2O(l) \quad (2)$$

As the ashes are left standing in open air, part of the reactive potassium oxide picks up carbon dioxide from the atmosphere to form potassium carbonate (eq 3).[14]

$$K_2O(s) + CO_2(g) \xrightarrow{\Delta} K_2CO_3(s) \quad (3)$$

Placing the ashes in water causes potassium carbonate to dissolve and hydrolyze, and the presence of potassium hydroxide (KOH) turns the solution alkaline. The overall reaction, showing the equilibrium between potassium hydroxide and potassium carbonate in water, can be described by eq 4.

$$K_2CO_3(s) + H_2O(l) \leftrightarrows 2KOH(aq) + CO_2(g) \quad (4)$$

If water is evaporated from the solution, solid potassium carbonate can be recovered as the chemical equilibrium shifts to the left side in eq 4.

Potassium carbonate hydrolyzes in water, producing alkaline pH. In an acidic medium, it forms carbonic acid, which readily breaks down into carbon dioxide and water. Potassium carbonate is also very hygroscopic, which means that this substance absorbs and holds the water from the surrounding environment.

Ashes are a complex system with a varied composition as a function of the burned biomass. According to Vassileva,[15] the water-soluble fraction of biomass ashes varies from 4% to 61%, and it is mainly inorganic compounds. Potash is the solid obtained from the aqueous extract from ashes. It is a mixture of salts, including carbonates, bicarbonates, other ions, and water-soluble compounds that resisted volatilization during the burning of the potassium-rich biomass, such as sodium carbonate, potassium sulfate, and phosphate.[16] To remove the remaining impurities, as well as moisture and organic matter that was not decomposed during the initial burning, the potash thus obtained was further submitted to a calcination step (heating to high temperatures in air or oxygen[17]).

To produce lye—the solution made out from the water-soluble fraction of ashes—and potash—the solid obtained after evaporating the lye—from biomass ashes in the laboratory, we propose an experimental activity that can be easily performed, using only the most basic apparatus. It can be used to stimulate and engage students both in learning chemical concepts as well as in discussions about the nature of science.

This activity can be used to illustrate concepts such as separation of mixtures, transformations of matter, chemical reactions, and pH of solutions, and it provides an excellent opportunity to discuss aspects of doing science in the classroom.

On Historical Contexts

Laboratory experiments are an integral part of effective scientific education. Only when students are afforded hands-on experience can the practical dimension of chemistry actually be conveyed in the classroom. However, it seldom happens that any historical elements are included in the experiments applied either in secondary or higher education.[18] By introducing the history of science into the chemistry teaching laboratory, students are encouraged to develop a multidimensional understanding of the nature of science[20,19] while, at the same time, developing the notion that scientific knowledge is essentially dynamic, undergoing constant development spurred by the ever-growing work of the scientific community. In addition to its intrinsic value as the foremost tool for presenting the nature of the scientific endeavor to students, historical experiments promote an ever more necessary interdisciplinary approach to the understanding of science as part of our cultures.[20] It is in this sense that Höttecke and Silva[21] have emphasized the necessity of the appropriate infrastructure. They point out that the regular teaching laboratory found in most schools or colleges may lack the facilities needed for implementing "historical" experiments. Therefore, the development of chemistry teaching with the use of "historical" experiments still necessitates a great deal of research before it can be seriously considered for widespread application,[22] especially bearing in mind the necessity for simpler and more affordable equipment and school infrastructures to counterbalance the enormous inequalities in science teaching.[23]

The experiment presented in this article has the advantage of only requiring readily available reactants that are easy to procure even in schools located in poorer locations, while it allows inquiry-based activities and other hands-on activities with alkaline solutions, such as soap production.[24,25]

■ MATERIALS AND METHODS

Producing Potash in the Laboratory

Before the laboratory session, the instructor may ask students to investigate in bibliographical sources some discussion about the presence of potassium in different plants. They may also be

Figure 1. Pictures of Potash obtained from Sibipiruna tree (*Caesalpinia pluviosa*, left), coconut tree (*Cocos nucifera*, center), and banana tree (*Musa paradisiaca*, right).

requested to select some plant material to be used in the activity.

For safety reasons, the instructor must perform the burning of the plant parts selected for the experiment to obtain the ashes. To make this step easier, the instructor can also ask for ashes in restaurants with a wood oven or barbecue grills. Knowing the origin of solid fuel, a specific type of wood or coal, it will still be possible to work on comparative studies. The steps were the following:

1. The students will weigh from 3 to 10 g of ashes (the instructor must check the yield previously to determine the amount since the potash content depends strongly on the source).
2. Add the ashes into a beaker along with 60 mL of water (this amount might be changed as a function of the water holding capacity of the ashes used in the experiment, and it must be tested previously to check the amount of time demanded in step 5 to evaporate all the water).
3. Stir the mixture continuously for 2 min and filter using a preweighed filter paper. The solution obtained after filtration is known as lye.
4. After the filtration, the filter paper containing the ashes should be left to dry on a stove set at 100 °C for 24 h, and its mass should be weighed after class for the yield calculation.
5. The filtrate was heated over a Bunsen burner until the water evaporates completely. The solid in the bottom of the beaker is the potash.
6. After the beaker with the potash crystals cools down, weigh and calculate the yield obtained.

The instructor should warn students strongly about weighing the paper filter, where the ashes dispersed in water will be filtered, and the beaker where the lye will be evaporated because the quantitative determination will be made through weighing by difference.

As an alternative to the drying of the filter paper (step 4) and the evaporation of the water (step 5), if the instructor has more time available for the experiment, a more sustainable alternative for drying or evaporation is the usage of air drying in a place with a lower chance of contamination by atmospheric deposition, like a closed fume hood or a laminar flow cabinet. Another sustainable alternative is using a smaller scale that could reduce both the energy for drying and evaporation and the time for the experiment. The only caveat is to be sure that the yield for the ashes is enough for the tests after potash production.

In Figure 1, we present a few examples of potashes to show the diversity of colors to be expected. These potashes were obtained from different sources: Sibipiruna tree (*Caesalpinia pluviosa*, from public pruning), coconut tree (*Cocos nucifera*, from public pruning), and banana tree (*Musa paradisiaca*, cut after harvest), respectively shown from left to right in Figure 1. Their yields were, on average, 8% for the Sibipiruna tree, 22% for the coconut tree, and 3% for the banana tree.

Testing the Resulting Potash

Presence of Carbonates. After weighing the beaker with the potash (step 5 of the procedure), a few drops of vinegar were added, and effervescence was observed.

Acid−Alkaline Character. The pH of the lye was measured using pH strips dipped in the lye solutions formed in step 3.

HAZARDS

To avoid contact with alkaline liquids, students and staff should wear protective goggles and gloves during the boiling of the lye due to the danger posed by a highly corrosive hot solution, by the flames, and by the collapse of the bubbles with the ejection of droplets. Any contact of this solution with eyes or skin is harmful and must be avoided. A thermal glove must be worn when handling glassware after being dried in the oven. The disposal of solid materials after the activity can be done in household waste. Solutions, once neutralized, can be disposed of in the sink. Potash is a mixture of potassium carbonate and oxide; the former causes skin and eyes irritation, and the latter causes severe skin burns and eye damage. Vinegar causes skin and eye irritation.

DISCUSSION

We performed the experiment with undergraduate classes of freshmen from the majors in Chemistry, Physics, Mathematics, and Biology. The students were divided into two classes, one with chemistry and physics students and one with mathematics and biology students. Each class had approximately 35 students. In addition, students were divided into groups of two or three students, totaling an average of 15 groups per class. The time spent performing the activity with each class was 2 h, although the experiment has a final step of drying after class.

Potash Production

Through the combustion of plants, the ashes contain a mixture of potassium oxide and carbonate. Potassium carbonate is obtained from the reaction of the oxide with carbon dioxide. Usually, there will be a predominance of carbonate due to its higher stability. By mixing the ashes with water, potassium carbonate and oxide will become soluble just as well as other soluble salts. Therefore, all soluble compounds will be in the filtrate. When the solution that has passed through the filter paper is evaporated, the solid formed will be potash.

We discussed the basics of extraction regarding the selective solubility of some compounds in water and the efficiency of extraction by controlling the weight of the potash obtained from the initial weight of the ash. In the proposed experiment, we obtained yields varying between 3 and 20 wt %. Additionally, the historical context fed a discussion in the

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laboratory about the role of the naturalist Fray José Marianno da Conceição Velloso to the technological development of potash production in Brazil. This discussion was not part of the report used to assess the efficiency of the experiment.

Regarding the yield, we had 75% of the correct answers and calculations. Since we have worked with freshmen students, we can conclude both that we have this concept well consolidated from the high school, and we have taught it meaningfully for 3/4 of the class.

Presence of Carbonates

By adding commercial vinegar in potash, we can observe the formation of carbon dioxide because potassium carbonate is a basic salt and reacts with acetic acid producing potassium acetate and carbonic acid, which quickly decomposes into carbon dioxide and water (eq 5).[26]

$$K_2CO_3(s) + 2CH_3CO_2H(aq) \rightarrow 2CH_3CO_2K(aq) + H_2O(l) + CO_2(g) \quad (5)$$

In the report, the students were asked to answer what equation could represent the effervescence observed after adding vinegar in the potash, and we have 67% correct answers. The main errors were the stoichiometry, the missing physical state of the substances, and some groups that represented the final product as the carbonic acid, without paying attention to the fact that the reaction did not represent the formation of gases, which is the observed phenomenon.

Acid−Alkaline Nature

When dissolved in water, potassium carbonate produces potassium hydroxide (eq 4), an Arrhenius base. In the proposed experiment, the students assessed the basic character of a potash solution by measuring its pH, which should be between 10 and 11. Most groups (85%) understood that the oxidation of potassium in plants produces potassium oxide which forms potassium hydroxide in water, giving the solution its basic character. A few groups (20%) also developed the idea that potassium carbonate could also be a source of a basic character because its hydrolysis produces a weak acid (H_2CO_3) and a strong base (KOH).

CONCLUSION

The proposed experiments provide a relevant context for teaching science with a historical context in a short time and a low demand for infrastructure, which is fundamental to the use in a wide range of situations. In addition, the historical episode involving potash production and its composition presents a range of content to be worked within the class and provides some scientific controversies, which we believe can be an essential strategy to make chemical education more attractive and motivating. The experiment also provided a context to teach students how to handle hazardous materials.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00896.

Handouts with notes for the instructors (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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