

# Study on sulfitation of fruits and berries: Methods of sulfitation and desulfitation

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**Abstract.** Sulfitation, the process of treating fruits and berries with sulfites, is a widely used method in the food industry to preserve their color, flavor, and overall quality. However, concerns regarding the health implications of sulfite consumption have prompted research into alternative methods and techniques for sulfitation and desulfitation. This paper reviews the current methodologies employed in sulfitation and explores emerging strategies for desulfitation, aiming to provide insights into safer and more sustainable practices in fruit and berry preservation.

## 1. Introduction

Fruits and berries are valuable sources of essential nutrients, antioxidants, and dietary fiber. However, their perishable nature makes preservation crucial for maintaining their quality and extending shelf life. Sulfitation, the addition of sulfites to these perishable products, has been a common practice for centuries due to its efficacy in inhibiting enzymatic browning and microbial growth. Nevertheless, concerns regarding sulfite allergies and its potential adverse health effects have led to the exploration of alternative preservation methods.

Sulfitation is a method of preserving fruits, berries and fruit and berry semi-finished products using sulfur dioxide SO<sub>2</sub> or an aqueous solution [1], sulfurous acid H<sub>2</sub>SO<sub>3</sub>, as well as salts of sulfurous acid, in particular sodium bisulfite NaHSO<sub>3</sub>. The use of sulfur dioxide for canning semi-finished fruit products and other similar products has been practiced in industry since the beginning of the 20th century. This method, which at one time played a certain positive role, should not be considered progressive at the present time. Working with sulfur dioxide is associated with a number of difficulties and dangers for workers servicing sulfitation plants. In addition, ready-made, sulfated, i.e., preserved by adding sulfur dioxide to them, fruit products are unsuitable and not harmless to the human body until the sulfur compounds are removed from them [2]. In addition, it is impossible to ensure completely complete removal of sulfur dioxide from fruit semi-finished products (the so-called desulphitation) before processing into other products mentioned above. The remaining small amount of sulfur dioxide in finished jams, marmalade and other fruit products in some cases gives them, although weak, but all same noticeable aftertaste [3]. Therefore, fruit products made from sulfated semi-finished products are not recommended for children [4]. In the national economy, measures are being taken for a gradual transition from sulfitation to the use of other methods that do not have the disadvantages listed above. One such method is canning using sorbates.

Sulfitation is well developed in the food industry, is sufficiently provided with both preservatives and equipment, and in addition to negative properties, it has a number of advantages (simplicity of the entire process, low cost, availability of implementation at any, even poorly equipped enterprises).

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## 2. Information about sulfur dioxide

Sulfur dioxide is a gas produced by the combustion of sulfur. It is 2.25 times heavier than air and in rooms or in vessels it is concentrated at the bottom, does not burn and does not support combustion. At temperatures below  $-10^{\circ}\text{C}$  and normal atmospheric pressure, sulfur dioxide turns into liquid. At normal room temperature, to transform it into a liquid state, it is necessary to increase the pressure. Sulfur dioxide dissolves easily in cold water. At  $0^{\circ}\text{C}$  you can obtain a solution with a concentration of 18% sulfur dioxide. But with increasing temperature, its solubility drops sharply; thus, at  $25 - 30^{\circ}\text{C}$  it is only about 7%. When heated to  $100^{\circ}\text{C}$  and boiling, the dissolved anhydride quickly evaporates, and this property is used for desulfation, i.e., freeing semi-finished fruit products and other products from preservatives.

Sulfur dioxide is toxic to all types of microbes, which die at an anhydride concentration in fruit and berry products of 0.1 - 0.2%. However, sulfur dioxide is also poisonous to humans. Sulfated products should not be directly consumed as food, as this may cause vomiting and other signs of poisoning [5.] When taking a large dose of sulfur dioxide, poisoning can be very severe. When inhaling gaseous sulfur dioxide, severe suffocation, irritation of the mucous membranes and other signs of poisoning appear. Therefore, when working with it, you must wear a gas mask.

Sulfur dioxide is produced in chemical plants. It comes in durable steel cylinders, where it is in liquid form under high pressure. In this form, sulfur dioxide is usually used for preserving fruit and berry purees or juices, as well as whole or crushed fruits and berries (the so-called pulp) in barrels of water. Sulfur dioxide, which has the same chemical composition, can be easily obtained on site by burning ordinary yellow lump sulfur. When sulfur burns, the resulting anhydride gas is used to fumigate fruits in boxes or baskets. In both cases, the effect of sulfur dioxide on fruits is exactly the same: it dissolves in cell sap and in the liquid surrounding the fruit, and when the concentration indicated above is reached, it causes the death of microbes [6]. Since the technique for using sulfur dioxide is not the same, it is customary to distinguish between two methods of sulfitation: dry fumigation of fruits with sulfur dioxide when burning sulfur and canning them with liquid sulfur dioxide from cylinders.

## 3. Fumigation of fruits with sulfur dioxide

The sulfur fumigation method is the simplest and can be used at poorly equipped processing plants or points. Whole fruits with fairly dense pulp are fumigated, mainly apples, pears, quinces, as well as apricots, cherries, cherries, and plums. The fruits are fumigated in special concrete, adobe or wooden chambers with gas-tight walls, as well as in tents made of thick tarpaulin impregnated with gas- and water-proof compounds [7, 8]. The cells should be convenient for ventilation and ventilation: with two doors and an exhaust pipe. For fumigation, pure cuttings or lump sulfur can be used.

Prepared clean fruits (for example, apples) in clean wooden lattice boxes are placed in chambers in stacks no more than 3 m high in a checkerboard pattern. Gaps of 2–3 cm are left between the boxes for better penetration of sulfur dioxide. Sulfur is lit in iron braziers on the floor of the chamber, on a special platform made of earth and sand (to avoid fire). A baking tray with sides is installed on the site, in which the sulfur is burned. Large lumps of sulfur are crushed into small pieces and scattered on a baking sheet, where a little dry firewood or kindling is placed in advance for kindling. The wood is set on fire and the sulfur also ignites. Per  $1\text{ m}^3$  of chamber capacity, 200 g of sulfur is burned. It is more convenient to burn sulfur in special metal braziers. Such braziers are made of roofing iron in the form of a cylinder with a diameter of 30–40 cm and a height of 50–70 cm. Air holes are punched in the walls of the cylinder, starting from 5 cm from the bottom to the top. Legs are attached to the bottom of the brazier, on which it is placed on the floor of the chamber on a layer of sand or on bricks. The sulfur is poured into a brazier, where small firewood or charcoal is placed in advance. The brazier is installed in such a way as to prevent the boxes of fruit from igniting. To do this, brick sides are installed around the site or iron sheets are installed on the sides so that splashes of burning sulfur do not fall on the tree. You can light the brazier outside the chamber, and when the sulfur lights up, bring it in and set it in place. After this, the chamber is tightly closed, and all the cracks in the walls and ceiling are coated in advance. If this is not done, the gas formed during the combustion of sulfur will quickly leak out, and the fruits will be insufficiently fumigated and, therefore, unstable in storage. The sulfur burns quickly, and the gas fills the entire chamber, saturating all the fruits in the boxes. In this case, fruits that are red or pink in color become pale, whitish with a yellow tint. To monitor the progress of fumigation, a small glass window is usually made in the wall of the chamber, on which several control fruits are placed [9]. Through the glass, the color change of these fruits is observed.

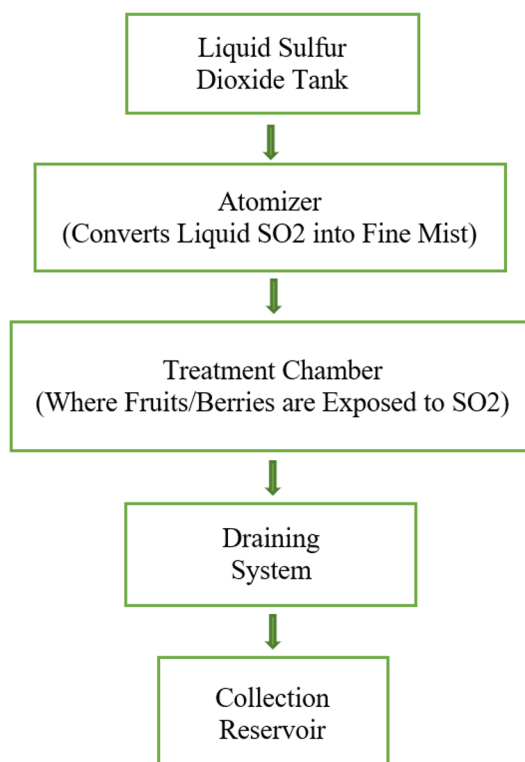
After combustion of the sulfur, the chamber is left closed for some time so that sulfur dioxide penetrates sufficiently into all boxes and has a preservative effect on all fruits. The recommended holding time for apples is 16 - 20 hours, pears - 12 - 15, cherries - 14 - 16, dogwoods - 12 -14, berries and apricots - 8 - 10 hours. Longer holding is not necessary, although it is not harmful to fruits. Unload the chamber carefully, remembering that it is filled with poisonous sulfur dioxide. First, open both doors and leave the chamber for natural ventilation for 2 - 3 hours, while checking whether all the sulfur has burned and what appearance the fruits have.

With good fumigation, apples and pears become pale, quite soft, break easily and have a noticeable smell of sulfur dioxide. To quickly free the chamber from sulfur dioxide, it is recommended to install a fan in it, which is turned on at the end of fumigation. When the ventilation is completed, you can enter the chamber, but be sure to wear a gas mask, since sulfur dioxide remains for a long time in the lower part of the chamber, between the fruits in the boxes. The fumigated fruits in boxes are taken out of the chamber and sent for storage [10].

Apples and quinces are stored in the same boxes, placing them in dense stacks, without gaps. The storage room must be clean, dry, without ventilation. Recommended storage temperature is from 0 to +10 °C. At this temperature, apples can be stored for 3–4 months. At higher storage temperatures or for longer periods of storage, stacks of boxes should be covered on top and sides with a thick tarpaulin to reduce gas losses. Other fruits cannot be stored in boxes after fumigation, as they lose some of the juice. Therefore, stone-shaped fruits and berries are placed in barrels and filled with water so that they do not wrinkle. Pears are stored in barrels without water.

#### 4. Sulfitation with liquid sulfur dioxide

The cylinder with sulfur dioxide is equipped with valves for releasing the anhydride. The valve on the cylinder opens using a handwheel. To prevent accidental damage to the valve during transportation or handling of the cylinder, there is a safety cap. When the cylinder is installed, the cap is removed and the valve can be opened and closed. A rubber hose is attached to the outlet, through which the anhydride exits the cylinder when the valve is opened. Since sulfur dioxide easily evaporates, it is necessary that it immediately enter the water upon exiting the cylinder, otherwise it will begin to evaporate, which will not only lead to large losses of anhydride, but is also harmful to the health of workers. Figure 1 illustrates the process of sulfitation with liquid sulfur dioxide.



**Fig. 1.** A simple diagram illustrating the process of sulfitation with liquid sulfur dioxide: *liquid sulfur dioxide tank* - contains the liquid form of sulfur dioxide; *atomizer* - converts the liquid sulfur dioxide into a fine mist, facilitating uniform application onto the fruits or berries; *treatment chamber* - where fruits or berries are exposed to the sulfur dioxide mist for sulfitation; *draining system* - allows excess liquid to drain away from the treated fruits or berries; and *collection reservoir* - collects any drained liquid sulfur dioxide for reuse or proper disposal

Liquid anhydride presses with great force on the walls of the cylinder; with increasing temperature, this pressure increases significantly (at 0° - 1.5 Pa, at +10° - 2.23, at +20° - 3.24, at +30 °—4.51, and at +40°—6.15 Pa). For safety reasons, storing cylinders with anhydride in the sun or generally at high temperatures is not allowed. When the valve is opened, liquid anhydride leaves the cylinder with high pressure into a space with normal atmospheric pressure and immediately turns into a gaseous state, trying to evaporate.

Sulfur dioxide is also used when fumigating fruits in chambers (as described above) instead of burning sulfur. In this case, instead of a brazier, the outlet end of a rubber hose connected to a cylinder containing anhydride is inserted into the chambers. The cylinder is placed on the scales, determining the amount of sulfur dioxide that needs to be released into the chamber. When such an amount of gas has been released (which is checked by placing weights or counterweights on the scales in the desired position in advance), the valve is closed. The calculation is relatively simple. We said above that when fumigating, 200 g of sulfur must be burned per 1 m<sup>3</sup> of chamber capacity. From 1 g of sulfur during combustion, i.e., when it combines with atmospheric oxygen, 2 g of sulfur dioxide is formed, therefore, per 1 m<sup>3</sup> of chamber capacity, 400 g of anhydride must be released from the cylinder. If, for example, the chamber is 5 m long, 4 m wide, and 2.5 m high, then its volume is 50 m<sup>3</sup> (5x4x2.5). Such a chamber requires 20 kg of anhydride (50x0.4). Fumigating fruits in chambers with sulfur dioxide from cylinders is more convenient and safer than burning sulfur.

But sulfur dioxide in cylinders is most often used for sulfitation of liquid fruit semi-finished products - puree, pulp, juice or fruits filled with water or juice [11]. In this case, the technique of its use is somewhat different. The anhydride from the cylinders is either directly released into juice or fruit puree, where it must completely dissolve, or a so-called working solution is prepared separately, i.e., the anhydride is dissolved in cold water, and then the resulting solution with a known concentration of sulfur in it is dissolved. Nitrous anhydride is accurately dosed into barrels or large containers of fruit products.

The first method, i.e. direct dissolution of the gaseous anhydride coming out of the cylinder in fruit products, is more economically feasible, since the fruit mass is not liquefied by adding water with anhydride dissolved in it and, therefore, the container is used more economically barrels, tanks or basins, in which sulfated semi-finished products are then stored. In addition, with this method, thermal energy is not consumed to evaporate the added water when jam or marmalade is made from sulfated semi-finished products.

The main method throughout the industry is to inject anhydride gas directly into the product. However, in order for the anhydride from the cylinders to be directly dissolved in the product, mixers and other additional equipment are required. Therefore, at some small, insufficiently equipped processing stations, it is necessary to use sulfur dioxide as a working solution.

To prepare the working solution, take a clean, dense, large (200-300 liters or more) barrel or vat and fill it with cold drinking water. A scale is placed nearby, on which a cylinder of anhydride is placed. A rubber hose connected to the outlet of the cylinder is lowered into the water so that its end is at the very bottom of the barrel or vat. Slowly open the valve on the cylinder until gas bubbles begin to come out of the hose. Then open the valve slightly more so that the number of bubbles increases, but so that all the bubbles have time to completely dissolve when passing through a thick layer of water. If the bubbles reach the top, it means that the gas evaporates into the air and is lost without benefit; in this case, the valve is slightly covered. Under normal summer conditions, at a temperature of 15-20°, no more than 5-6% of sulfur dioxide can dissolve in water. Therefore, in practice, the working solution is most often prepared at this concentration. It is advisable to have a more concentrated solution so that less water gets into the sulfated products.

**Table 1.** The relationship between the specific gravity of an aqueous solution of sulfur dioxide and its concentration

Specific gravity of solution, g/ml at 15°	Solution concentration, %	Specific gravity of the solution, g/ml at 15°	Solution concentration, %	Specific gravity of solution, g/ml at 15°	Solution concentration, %
1.0181	3.25	1.0248	4.50	1.0315	5.75
1.0194	3.50	1.0261	4.75	1.0328	6.00
1.0206	3.75	1.0275	5.00	1.0340	6.25
1.0221	4.00	1.0289	5.25	1.0353	6.50
1.0234	4.25	1.0302	5.50	1.0365	6.75

The calculations for preparing the working solution are also simple. If a solution is prepared with a sulfur dioxide concentration of 5%, and 350 liters of water are poured into a vat, then  $(350 * 5)/100 = 17.5$  kg of anhydride must be dissolved in it. Set the counterweight to 17.5 kg less than the initial weight of the cylinder; Thus, a sufficient amount of gas is released from the cylinder and enters the water. However, it may happen that part of the gas passed through the layer of water and, without having time to dissolve in it, was lost into the atmosphere. Then the working solution will have a concentration lower than calculated. If this is not taken into account, sulfated semi-finished products may have an insufficient concentration of sulfur dioxide and they will begin to deteriorate due to fermentation.

Therefore, the actual concentration of the working solution should be systematically checked before use. The concentration of sulfur dioxide in water can be checked by the specific gravity of the solution using a hydrometer and a glass cylinder. Having determined the specific gravity, find the concentration of the working solution according to Table 1.

It is advisable not to store the finished working solution for a long time, but to use it immediately; if necessary, store in a covered container in a cool room. It is impossible to store the working solution, as well as all sulfated semi-finished products, in iron containers, since sulfur dioxide reacts with iron, causing severe rusting. All valves, pipes and other parts of equipment and equipment in contact with the solution must be made of brass or other non-corrosive materials. When using a working solution, calculate the amount that should be added to a barrel or other container with sulfated semi-finished products in order to obtain a preservative concentration that provides a preservative effect. Thus, when canning applesauce, the concentration of sulfur dioxide in it should be from 0.12 to 0.18%, i.e. from 1.2 to 1.8 g/l. If we take an average concentration of 0.15%, i.e. 1.5 g/l, then a barrel of puree with a capacity of 200 liters will require  $200 \times 1.5 = 300$  g (0.3 kg) of pure sulfur dioxide or a working solution with a concentration of 5, 5%  $(0.3 \cdot 100) / 5.5 = 5,450$  l.

The current technological instructions establish the following doses for adding sulfur dioxide to sulfated products (in% of the weight of the product) is shown in Table 2.

**Table 2.** Doses for adding sulfur dioxide to sulfated products (in% of the weight of the product)

Apple, cherry plum, plum puree	Berry puree	Melon, peach, apricot puree, etc.	Whole cherries, black currants	Whole apricots, plums
0,1—0,18	0,1—0,15	0,12—0,20	0,2	0,15

When certain fruits, berries or products are sulfitized, their usual technological preparation is first carried out - washing, inspection and removal of all defective specimens.

If whole fruits and berries are sulfated in an aqueous solution, then these fruits are loaded into barrels and the calculated amount of anhydride working solution is poured into them. In this case, the calculation is made for the total amount of product, including fruit and liquid, since the anhydride is distributed evenly throughout the entire volume. Usually, when sulfiting (for example, cherries), a 1% solution of sulfur dioxide is poured into a barrel filled with fruits in an amount of 20% by weight of the fruit. This ensures the required preservative concentration of 0.2%. The barrels are sealed so that the solution is completely distributed on the surface of the fruit. After this, the barrels are left for 3-4 days. When the fruits have settled somewhat, the barrels are opened, sulfated fruits from other barrels are added on top, the sheaf is sealed and left for storage. Thus, relatively little water is added to the barrels with fruits.

When sulfiting fruit purees, the washed fruits are scalded in shaft scalders or digesters, and when they are sufficiently softened, they are passed through rubbing machines. Hot puree cannot be sulfated, because most of the anhydride introduced into it will evaporate almost immediately and such puree after cooling will no longer be shelf stable. Therefore, the puree is first cooled to a temperature of 35-40°, and preferably lower. The puree is then mixed with the anhydride solution in a mixing apparatus. The simplest mixer is a large (300-500 l) wooden vat or barrel mounted horizontally on a rotating shaft. On the side surface of the vat there is a tightly closing hatch, through which cooled puree and a working solution of sulfur dioxide are loaded in its upper position. Then the hatch covers are closed and the barrel is rotated using a motor for 2-3 minutes. During this time, the puree is sufficiently mixed with the anhydride; Such puree can be poured into tightly sealed barrels for further storage.

The industry uses more advanced and high-performance mixers for sulfitation of fruits and purees. Instead of a working solution, pure sulfur dioxide is introduced into the puree. For large production volumes, the use of barrels for packaging sulfated semi-finished products is irrational and causes many difficulties. Therefore, large factories use large containers for storing sulfated products: wooden boards, the same as for fermenting vegetables, or concrete rectangular or cylindrical basins that can hold 20-50 tons or more. However, if the sulfated fruit mass comes into direct contact with concrete, the latter can be destroyed by acid and other substances, as a result of which sand and even small pebbles can get into the product. To prevent this from happening, the inner walls of concrete containers are coated with a layer of chemically resistant compounds.

For several decades, concrete containers coated with various protective compounds have been used for sulfitation [12, 13]. However, special attention should be paid to one very important circumstance: the instructions provide for several compositions of “resins” for such coatings (almost all compositions include bitumen). According to the latest medical research, bitumen can have a harmful effect on the human body if food products have been in contact with it. Therefore, the use of coatings containing bitumen is currently prohibited. New formulations are being developed. Until their introduction into practice, one should refrain from using bitumen-based tars; If necessary, consult with sanitary inspection institutions. You need to focus on the use of wooden boards, and use paraffin or liquid glass to cover concrete containers.

## 5. Desulfitation

Sulfitation is carried out during the fruit harvest season, i.e. in summer and autumn, but the processing of sulfated semi-finished products can continue for many months and even a year. During this entire time, sulfated semi-finished products are reliably preserved under the influence of sulfur dioxide.

All sulfated semi-finished products, i.e. mainly purees and pulp, as well as whole fruits, are intended for the subsequent production of new fruit products from them - jams, marmalade, fillings for confectionery products, syrups, jellies, etc. In all cases, the first and most important operation is desulfitation, i.e., removal of sulfur dioxide from semi-finished products. The presence of this preservative in the listed finished products is not allowed, with the exception of insignificant traces (thousandths of a percent), which are practically impossible to remove. Desulfitation is based on the fact that when heated to the boiling point, sulfur dioxide quickly evaporates. The traces remaining in the product are sulfurous acid, bound to some of the components of the product by strong chemical bonds [14]. To desulphate, the semi-finished product is placed in a two-body boiler or other cooking or evaporating apparatus and heated at boiling for 15 - 20 minutes.

When carrying out desulphitation, one must remember that sulfur dioxide is poisonous to humans. Released from sulfated semi-finished products during the desulfitation process, it can enter the workshop and cause serious poisoning of workers. Therefore, all devices in which desulfitation is performed must be equipped with sufficiently powerful local exhaust ventilation. In addition, the workshop must have general supply and exhaust ventilation to remove from the room traces of sulfur dioxide released from barrels with sulfated semi-finished products or entering the workshop in other ways.

All workshop equipment, especially parts in contact with the product, must be made of non-corrosive materials. Iron fittings, all kinds of pipelines and the pipelines themselves for water, steam and other communications, especially ventilation pipes and ducts made of sheet iron, suffer greatly from the effects of sulfur dioxide. Where it is impossible to replace iron with other materials, the surfaces of pipes, ventilation ducts, etc. should be carefully coated with a layer of drying oil and painted with strong, durable paint.

Below is the major desulfitation Techniques:

- a) Washing and Rinsing: One of the simplest methods of desulfitation involves washing and rinsing treated fruits and berries with water. This process helps remove surface residues of sulfites, reducing their concentration to safe levels. However, thorough rinsing is essential to prevent sulfite residues from leaching back into the fruit or berry tissue.
- b) Enzymatic Desulfitation: Enzymes such as sulfite oxidase can catalyze the conversion of sulfites to sulfates, which are less reactive and generally considered less harmful. Enzymatic desulfitation offers a promising approach to reducing sulfite residues in treated fruits and berries while minimizing nutrient loss and preserving product quality.
- c) Atmospheric Modification: Controlled atmospheric storage techniques, such as modified atmosphere packaging (MAP), can facilitate the desulfitation process by promoting the oxidation of sulfites to sulfates. By adjusting the oxygen and carbon dioxide levels in the storage environment, MAP can accelerate the degradation of sulfites while maintaining the freshness and appearance of the produce.

## 6. Conclusion

Sulfitation remains a prevalent method for preserving the color, flavor, and shelf life of fruits and berries in the food industry. However, concerns regarding sulfite residues and their potential health implications have prompted research into alternative sulfitation and desulfitation techniques. By exploring methods such as enzymatic desulfitation and atmospheric modification, researchers aim to develop safer and more sustainable practices for fruit and berry preservation. Further studies are warranted to optimize these techniques and ensure their efficacy and feasibility in commercial applications, thereby addressing consumer concerns and promoting the production of high-quality, minimally processed fruit and berry products.

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