Development of a monolithically 3D printed reciprocating piston pump for HPLC

A Senior Honors Thesis

Submitted in Partial Fulfillment of the Requirements
for Graduation in the Honors College

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The College at Brockport
December 14, 2020

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ABSTRACT

There has been much research in recent years aiming to make scientific instrumentation more accessible. An increase in accessibility has many benefits including reduced costs and expanded opportunities to learn about instrumentation. 3D printing of scientific instrumentation provides an option that is cheap and customizable. This study follows the development of a 3D-printed ball check valve to be implemented in a reciprocating piston pump such as one used for high performance liquid chromatography (HPLC). The reciprocating piston pump is just one of several types of positive displacement pumps. Seven types of positive displacement pumps will be described, as well as some unexpected applications in the sciences or every day life. The valve designed in this study was designed using OpenSCAD and printed using a Prusa i3 MK2S 3D printer. Ball check valves with both a spherical and conical design were designed, and early qualitative tests point towards the conical design being desirable. Future work includes the design of a reciprocating piston, the implementation of the piston along with two check valves to create a complete reciprocating pump, and quantifying pressures achieved by the pump.

INTRODUCTION

Increasing Accessibility

Making scientific instrumentation more available has proven to be a goal for many researchers in recent years. There are several factors that contribute to increasing accessibility, including lowering costs of devices and distributing information for creating instrumentation in an open-source format. Open-source instrumentation can take the form of instructions and files necessary for one to create their own devices. These files can be edited by the user to fit whatever parameters they need, creating personalized instrumentation for any situation. Improvements can also be made upon these designs and shared, resulting in an ever-evolving and improving piece of instrumentation or equipment that the internet community contributes to. 3D printing is a fabulous way of drastically lowering costs and allowing others to improve upon designs, since STL files are commonly shared to the internet and users are able to edit those files. The materials for 3D printing are also relatively inexpensive, and 3D objects are quick to produce—typically needing anywhere from a few minutes to a few hours.

A powerful combination in the area of accessible instrumentation is a Raspberry Pi combined with Arduino. "Homemade" instrumentation generally includes some electronic component that needs to be controlled using a computer and some software. A Raspberry Pi is an ideal computer, due to its extremely small size and ability to be used nearly anywhere as long as there is some sort of display that it can be connected to. Arduino is an open-source hardware that uses a programmable microcontroller so one can run their electronics as they had intended. The programming software is available as a free download online, so that anyone is able to use it and even create their own custom software to use with the microcontroller[1].

The Arduino microcontroller can be used in many unexpected devices. Glasscott et. al. developed a two-electrode potentiostat that utilizes an Arduino microcontroller. Their so-called SweepStat consists of an Arduino Teensy 3.2 interface and a printed circuit board with custom developed circuitry. This device costs $55 to make, where a commercial three-electrode potentiostat can cost anywhere from $4,000 to $40,000. Moreover, the SweepStat can perform cyclic voltammetry, linear sweep voltammetry, chronocoulometry, and chronoaperometry, which are more experimental capabilities than some other comparable, yet slightly more expensive to produce, low-cost potentiostats[2]. The use of the SweepStat is exciting for one who is interested in electrochemistry. Research in electrochemistry or even learning how it works in a lab setting is something that might not be as readily available to students because of the cost of a commercial potentiostat. Further, an institution that does have a potentiostat is likely to only have one, limiting the number of students who are exposed to the instrument at any given time. At $55, the SweepStat provides a more accessible alternative to encourage electrochemical education. Another perk is that since one must assemble the SweepStat themselves, they will get the opportunity to learn more about electronics.

HPLC

The technique that was the inspiration behind this research is HPLC, or high performance liquid chromatography. At its core, HPLC is simply a separation method. Chromatography takes advantage of different properties of molecules, such as size, polarity, or charge, to separate them from one another in a mixture. HPLC is one of the most widely used chromatography methods because it can be used to separate a wide variety of substances, including organic, inorganic, and biological molecules. The main components of HPLC that allow the separation to take place are the column and the solvent reservoirs. The solvent reservoirs house different liquids of varying polarity indexes. These solvents are combined together in specific ratios that make up the mobile phase, or the element which moves through the column and carries the components of the mixture being studied. Some common mobile phases include cyclohexane, ethanol, acetonitrile, and water[3].

The column is where separation takes place. Here is where the stationary phase is located, or the solid element that does not move through the column. Both the stationary phase and mobile phase can be chosen depending on the type of separation that is desired. Adsorption or normal phase chromatography takes advantage of the molecules' solubilities in water, while reverse phase takes advantage of molecules' solubilities in organic solvents. Ion exchange chromatography utilizes the net charge on molecules to achieve a separation while size exclusion chromatography separates molecules on the basis of size[4].

More specifically, normal phase chromatography uses a highly polar stationary phase and a nearly nonpolar mobile phase to elute different components in order of increasing polarity; since the mobile phase is nonpolar, nonpolar molecules will elute off the column more quickly than polar compounds that adsorb to the polar stationary phase. Reverse phase chromatography uses the exact opposite setup: the mobile phase has high polarity while the stationary phase is more nonpolar, and compounds elute off the column in order of decreasing polarity[5]. These two methods are useful in separating organic molecules such as hydrocarbons, oxygenated hydrocarbons, or any functional groups that might add polarity to the molecule. The stationary phase of ion-exchange chromatography may be chosen to be either negatively or positively charged; if the stationary phase is chosen to be negatively charged, positively charged ions contained in the mobile phase will be attracted to the stationary phase and only negatively charged ions will elute off the column, and vice versa[4]. This method is useful in selecting for analysis of only cations or anions, and may be applied to inorganic molecules. The stationary phase of size-exclusion chromatography is chosen so that small molecules are trapped, only allowing large molecules to pass through the column[4]. The type of molecules that readily come to mind for this type of separation are biological molecules, such as proteins, nucleic acids, steroids, or hormones.

HPLC requires high pressures to operate—up to 6,000 psi. As will be discussed, this high pressure is typically provided by a reciprocating piston pump. However, some research labs are working to find innovative ways to design different types of pumps that can perform the same function using a completely different mechanism. Wang et. al. developed a high-pressure pump for HPLC that functions as part of a microfluidic “lab-on-a-chip” device. While reciprocating pumps require a motor to mechanically move the piston and displace the fluids, the pump designed by Wang depends only on electroosmosis. The pump consists of a silica surface that becomes negatively charged, attracting cations to form a positively charged layer close to the silica surface. An electric field is applied, causing the movement of cations which creates an electroosmotic flow, and no motor is necessary[6].

Only one electroosmotic cell is not enough to generate high pressures, but Wang et. al. found that as the number of electroosmotic cells increases, so does the maximum output pressure. When the pump was incorporated into a lab-on-a-chip device to perform HPLC, the results obtained from the “homemade” HPLC and a commercial Agilent 1200 HPLC are astonishingly comparable. The efficiency and resolution of both were similar in several trials and most peaks were common between the two, which was expected since both used the same column and same solutions[6]. Still, this result is promising for the end goal of this study—to create a 3D printed reciprocating piston pump that can function as a pump for HPLC. Wang et. al. created a pump that was electrochemically powered whereas the pump in this study will be mechanically powered, but they did successfully reach their goal.

Positive Displacement Pumps

The pump that supplies the high pressures needed for HPLC is a reciprocating pump. This is one class of what are called “positive displacement pumps,” or pumps which work to mechanically move fluids through the desired system. A positive displacement pump is useful in situations that require high pressures and steady flow rates. These pumps can also be used to move fluids of higher viscosity[7]. They can also have small internal volumes, some achieving internal volumes on the scale of microliters. In each of the different types of positive displacement pumps, there is some moving element that displaces the water. The motion can either be a back-and-forth which changes the volume of water in the pump or the water can be moved through the rotation of some element—these two designs are the two major classes of positive displacement pumps: reciprocating and rotary pumps.

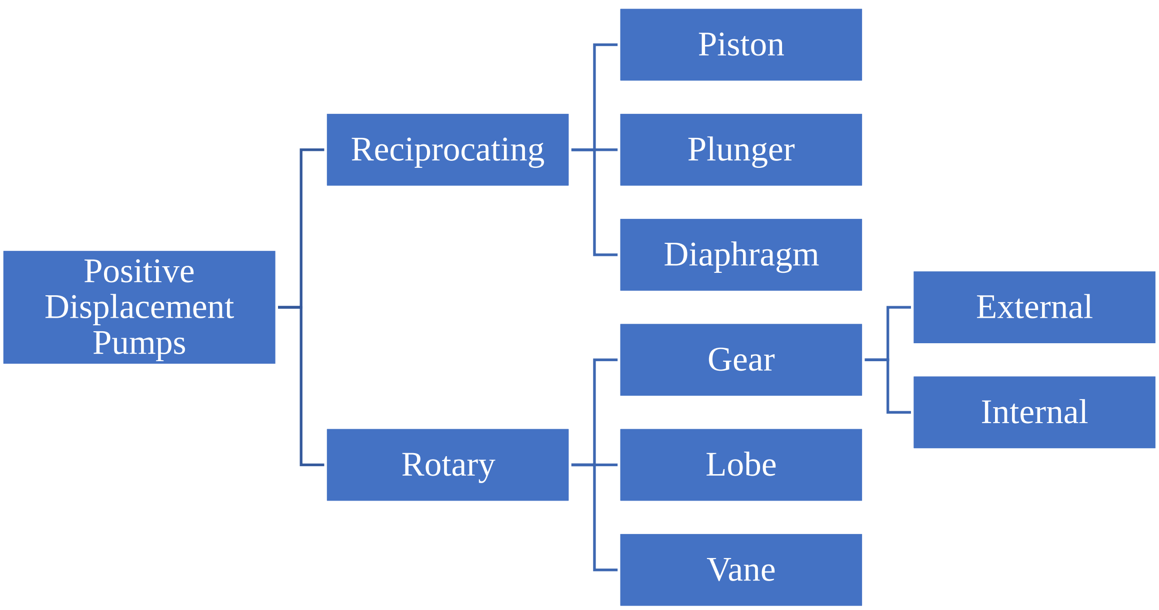


Figure 1. The reciprocating piston pump, which was the goal for this study, is only one of several types of positive displacement pumps.

Figure 1. shows the seven different types of positive displacement pumps that will be discussed presently and how they are classified. All of the different mechanisms will be described in terms of their design and function, as well as some examples of how these pumps are

implemented in recent research in hopes to illustrate the versatility of positive displacement pumps.

Each of the three types of reciprocating pumps all work by the same mechanism. They all involve a fluid-filled chamber, with check valves on either side hooking up to the inlet and outlet. There is some moving element (piston, plunger, or diaphragm) that works to alter the physical space inside the chamber. The "suction" stroke increases the area of the chamber, which decreases the pressure of liquid inside the chamber and draws in more liquid through the open inlet valve. Then on the "compression" stroke, the inlet valve is pushed closed and the outlet valve is pushed open. The space inside the chamber decreases, increasing the pressure of the fluids inside the chamber and forcing the fluids to move along through the outlet.

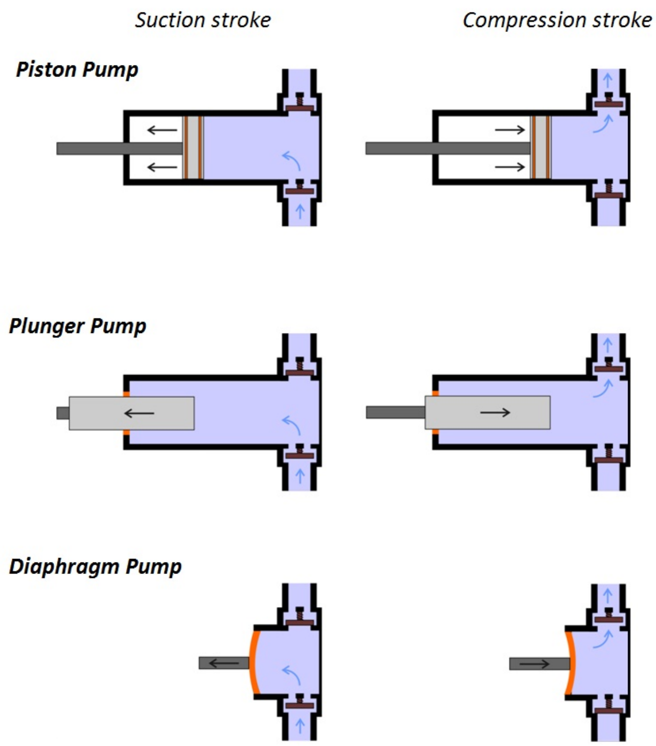


Figure 2. Basic mechanism of each of the three reciprocating pumps[7].

Figure 2. illustrates the funciton of the reciproacting pumps as previuosly described. The reciprocating piston pump utilizes a tight-fitting piston, normally paired with an o-ring to ensure a leak-proof seal, to move fluids through the device. The reciprocating piston pump is the kind normally found in an HPLC, but there are countless other applications of this kind of pump. One unexpected use of these pumps is found in the hydraulic system of civil aircrafts, or non-military planes that carry passengers. These pumps are the component that supplies mechanical power to be converted to hydraulic power. The hydraulic power is then used in the components of the aircraft that work to keep the flight of the plane steady, and the landing and breaking mechanisms[8]. The reciprocating piston pumps used in aircraft hydraulic systems are considerably more complex than pictured in Figure 2. The main features of the system consist of several pistons attached to a plate that rotates on its axis at an angle, which is called the yoke, and an attenuator at the outlet port[9]. The yoke adjusts the displacement of the pistions and ensures that each individual piston has its own position, but that all of the pistons move at the same rate. The attenuator is a vital component of the pump system because this helps to regulate the flow leaving the pump. If the attenuator were not included, the motion of the pump would result in a pulsing flow.

One potential problem that can be faced when using a reciprocating piston pump is the generation of unwanted heat. There are a lot of moving components within a system containing a reciprocating piston pump, and therefore a lot of frictional forces at work resulting in an increase in temperature. The piston can become poorly lubricated in its cylinder, resulting in a rise in temperature and therefore decreasing the ablility of the system to efficiently dissipate heat. The temperature change in an aircraft as the pump increases from 3000 psi to 8000 psi can be as drastic as an increase of 70°C, as the system increases from 110°C to 180°C[10]. Therefore, a cooling system must be in place in the event of unwanted heat generation that alters the function or efficiency of the pump.

Plunger pumps are similar to piston pumps, but the plunger is loose in the cylinder whereas the piston has a tight seal. There are some advantages of using a plunger pump over a pistion pump, such as the ability of plunger pumps to achieve higher pressures, the fact that the plunger does not touch the walls of its chamber, and that plungers can move faster than pistons because they have a smaller size and mass[11]. An important characteristic of the plunger is that its length must be much greater than its diameter. A common design of reciprocating plunger pumps is the plunger being connected to a crank shaft. The turning of the crank shaft induces the reciprocating motion of the plunger that drives the displacement of the fluids.

Velikanov et. al. present some data on WEIR SPM TEM 2500 and QEM 3000 plunger pumps, which are plunger pumps arranged in a system of three (TEM 2500) plungers or five (QEM) plungers. Specifically, the pressures achieved by each of the pumps is presented when the plunger is 101.6, 114.3, or 127 mm in diameter. The maximum pressures achieved by the three different diameter plungers are 151, 119, and 97 MPa, respectively, for the three-plunger system and 152, 119, and 97 MPa, respectively, for the five-plunger system[11]. This means that both systems can reach pressures up to around 21,900 psi. A third pump, the LEWA high-pressure plunger pump, achieved pressures up to 350 MPa, or around 50,700 psi[11]. An interesting relationship is that as the diameter of the plunger decreases, the maximum pressure attainable by the system seems to increase. This may be because the plunger is able to move faster as its size and mass decreases.

Velikanov et. al. investigated the relationship between the output pressure and the pump supply, or the amount of fluid supplied to the pump measured in L/min. They found that, generally, the output pressure of a plunger pump does not depend on the pump supply and remains constant as pump supply increases, until a certain point where the pump supply exceeds the capabilities of the pump. From there, output presssure of a plunger pump decreases with increasing pump supply[11]. So, there is an optimal range for the performance of reciprocating plunger pumps, but as long as the pump operates within that range, high pressures can be reached.

Diaphragm pumps make use of a flexible diaphragm that expands and contracts the volume of the pump. Fluids are drawn into the cylinder upon expansion and expelled through the oulet upon contraction. Since diaphragm pumps are sealed, there is a significantly lower chance of leaking fluids and the pump may even have a longer lifetime. This makes these pumps ideal for pumping hazardous materials[7]. However, diaphragm pumps are extremely versatile. A study done in 2000 used a diaphragm pump as a main component in a sampling device. Doskey et. al. designed an automated sampling device to measure the non-methane organic compounds that are found in ambient air samples. The measurements were taken in an evacuated canister and a Neuberger Viton diaphragm pump was used to pressurize the canister with ambient air[12]. This demonstrates the ability of reciprocating pumps, specifically diaphragm pumps, to effectively move fluids other than liquids.

The second class of positive displacement pumps, rotary pumps, operate by a different mechanism than reciprocating pumps. All rotary pumps utilize a rotating chamber that traps the fluid, which is mechanically moved through the device to the outlet.

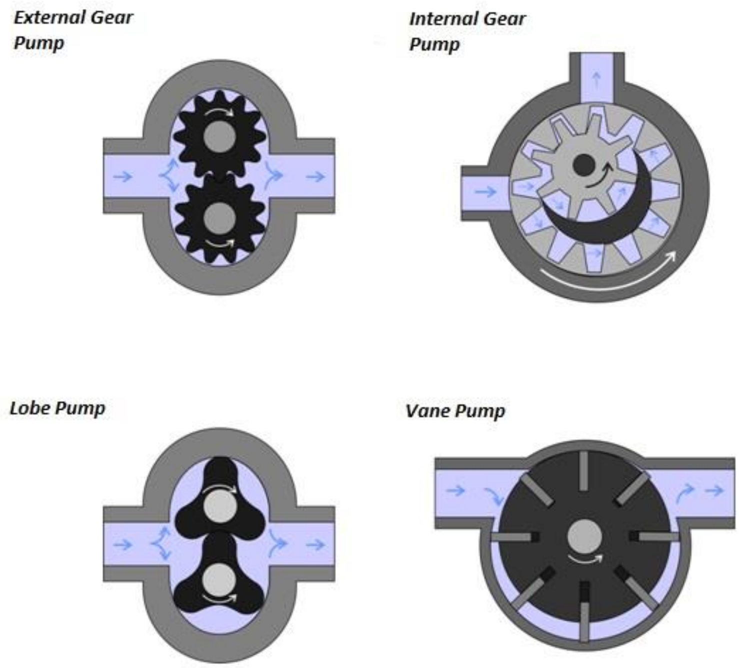


Figure 3. Design of rotary pumps. Each rotary pump contains a rotating element and a chamber which holds the fluid being pumped[7].

Figure 3. displays the design of four kinds of rotary positive displacement pumps. In the external gear pump, there are two interlocking gears within the housing of the pump. Fluid gets trapped between the space between the teeth of the gears and the inside wall of the pump. The trapped fluid moves along until the teeth of both gears meet, where the fluid is squeezed out of the space between the teeth and is forced through the outlet. The external gear pump is one of the most commonly used pumps in hydraulics systems, and several factors contribute to their popularity. These pumps can achieve high pressures, are relatively simple and inexpensive to construct, and can be customized to be optimal for the system in which one of these pumps is implemented[13].

The internal gear pump operates similarly to the external gear pump, but the design consists of a smaller gear that rests inside of a larger gear. The two are separated by a crescent element and meet at one point. The fluid gets trapped in between the space between the teeth of the gears and the wall of the crescent. The trapped fluid is moved until the point at which both gears meet, where the fluid is then forced out of the outlet. Another difference between the external and internal gear pump is that the internal gear pump is more costly to produce, but has less wear and longer lifetimes than the external gear pump. Internal gear pumps are ideal for pumping fluids of high viscocity, and they have a few unexpected applications as a consequence. Sanitary internal gear pumps are utilized by food production plants to move pastes and sauces, such as tomato paste or chocolate sauce. They are also an important component in engines of automotives[14].

The lobe pump is similar to the gear pump, with one key difference—the two moving elements of the lobe pump do not touch each other at some point. Where the motion of one gear causes the movement of the other, each lobe is controlled independently of the other by some mechanical element. The fluid moves through the device, getting trapped between the walls of the pump and the space between each rotor. The fluid is then pushed to the outlet when the rotor meets the other lobe because there is such a small space between the two that the fluid is forced out of that space. Lobe pumps are often not used for high-pressure applications, and instead operate at low pressures with high flow. Some examples of devices where lobe pumps are utilized include blowers, vacuums, and in the food industry[15]. The lobe pump therefore has a unique and very specific design, and a different function than other positive displacement pumps previously discussed. Where most other pumps have high output pressures, lobe pumps generally operate at low output pressures, adding to the variety and versatility of the uses of positive displacement pumps.

The vane pump consists of a rotor that has spaces for vanes to freely slide in and out. Since the rotor is not exactly in the middle of the chamber, the vanes are constantly moving pressed flush along the walls of the housing, moving in and out of the rotor as necessary. The fluid is trapped in a chamber formed between the walls of the housing and the space between each vane, and is forced out through the outlet once the vane reaches the point where the rotor is closest to the walls of the housing. An advantage of using a vane pump is that the vanes are constantly moving against the walls of the housing, so it essentially automatically adjusts to any wear that might happen. Once the vanes wear down to the point where the pump's function is affected, the vanes themselves are very easy to remove and replace[15].

Positive displacement pumps are therefore extremely versatile and have applications in many areas of science—including commercial instrumentation, such as HPLC, and instrumentation designed in the lab, such as the sampling device by Doskey et. al.— and day-to-day life, such as in automobiles and aircrafts. The advantages of the different kinds of positive displacement pumps vary, ranging from high pressures attainable, to reduced potential of leaks; from inexpensive production to the ability to be optimized for nearly any system. Rotary pumps and reciprocating pumps work by two very different mechanisms. Rotary pumps trap fluids within chambers that move through the device to the outlet, where reciprocating pumps displace the fluid through suction and compression, and flow is allowed by the operation of check valves. The development of my own check valve is presently discussed.

METHODS

The design process of the ball check valve consisted of writing code, printing on a 3D printer, and qualitative testing. All prints were designed using OpenSCAD software. All prints were made using a Prusa i3 MK2S 3D printer. The extruder was heated to 212℃ and the printer bed was heated to 57℃ and the layer height was set to 0.20 mm. Polylactic acid (PLA) filament (Gizmodorks) was used for all prints. 3D objects were not subjected to any treatments after printing and were tested qualitatively using deionized water manually flowed through 2.79 mm pump tubing (Fisherbrand). No quantitative tests were performed using the designed check valves.

RESULTS AND DISCUSSION

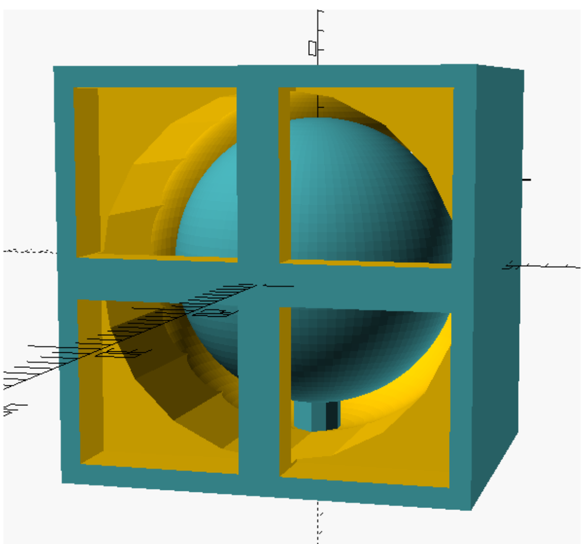
The first step in the design of the ball check valve was to ensure that a freely moving ball could be successfully printed inside a cavity of a larger object. Since the goal was to create a monolithic object, a sphere was attached to a support pillar resting inside a spherical cavity of a cube. The layer height for this print was 0.35 mm.

Figure 4. Cross section of initial design for the ball in socket. Dimensions: sphere—15 mm diameter, support pillar—1 mm diameter, spherical cavity—18 mm diameter, cube—20x20x15 mm. The handle on top was merely to help snap the ball off the support pillar.

Figure 4. displays the initial ball in socket design, as viewed in the Prusa Slic3r. The print was successful in confirming that a free-floating ball could be achieved through a monolithic design, but failed in most other areas. For one, the layer height was too thick, resulting in wispy strands of filament towards the bottom of the ball that broke off when the ball was moved around. Next, the diameter of the support pillar was slightly too narrow to be reliably printed within the

constraints of the printer and the chosen layer height. Finally, the entire unit was several times larger than needed for the desired size of the finished valve.

The next step of the design process involved implementing this ball-in-socket design in a check valve. The necessary criteria of the design included a free floating ball which when in one position allowed the flow of liquids through the valve and blocked the flow of liquids through the valve in the other position. The intuitive design was to have one side of the valve open to flow, but with some element to contain the ball within the space of the valve. The other side should have had some space in which the ball created somewhat of a seal with the walls of the valve, prohibiting the flow of liquids when the ball was in that position.

a. 

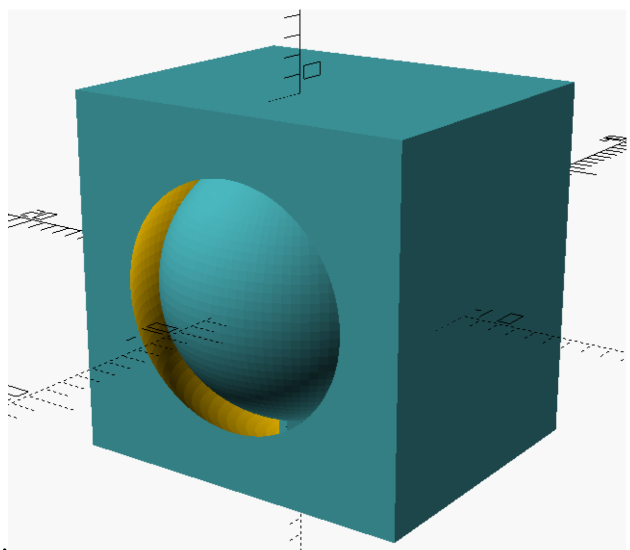
b. 

Figure 5. Initial valve design. **a.** This side of the valve permits flow; ball moves through a cylindrical channel and is kept in place by the cross. **b.** This side of the valve prohibits flow; ball becomes nestled in a spherical cavity, creating a seal.

The goal of the print in Figure 5. was to determine if a monolithic valve could be designed and successfully printed. Again, this print was made with a 0.35 mm layer height, so the bottom of the ball was essentially printing over air and the resulting print had wispy pieces of filament instead of a nice sphere shape. Since the use of support material was not desired, this same object was reprinted with 0.20 mm layer height which alleviated the problem of printing over air and resulted in a smooth sphere. The 0.20 mm layer height was used for all subsequent prints.

After the valve in Figure 5. was successfully printed, the smallest possible size of the valve was determined. The initial design consisted of a sphere 15 mm in diameter inside of a 15x20x20 mm cube. The size of the cube was halved so that it measured 10 mm in the y and z directions, and the rest of the dimensions were adjusted accordingly. This size valve was printed with no problems, so the size of the cube was halved again so that it measured 5 mm in the y and z directions. This size proved to be too small to print successfully given the limitations of the 3D printer. Therefore, a cube measuring 7.5 mm in the y and z directions was determined to be the optimal size. To test the valve's performance, the valve was implemented in a T-shaped channel.

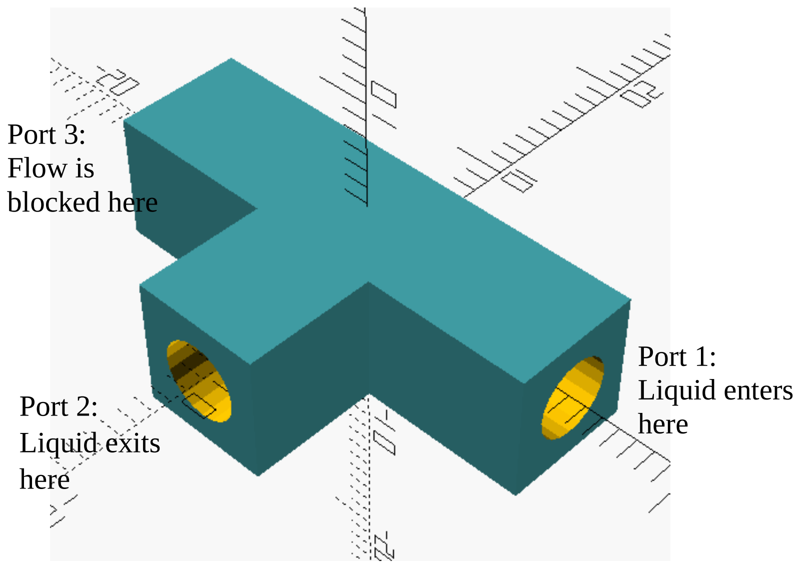


Figure 6. Check valve should result in a specific flow of liquid.

Figure 6. shows the design of the T-channel created to test the performance of the valve. Tubing was attached to ports 1 and 2. A plastic syringe was used to manually deliver deionized

water to port 1. The ball check was located in port 3 and no water should have leaked out of that port, rerouting the flow of water out of the channel exclusively to port 2. The flow of the water delivered was controlled simply by pressing the syringe with different pressures or speeds, for example, slowly emptying the syringe created a gentle flow of water while pressing down harder created more water pressure in the device. The expected outcome was not observed and port 3 was leaky even under conditions of higher water pressure.

A slight modification was made to the valve where instead of the ball nestling in a spherical cavity to block flow, the ball would be pushed into the tapered end of a conical cavity within the valve.

a. b.

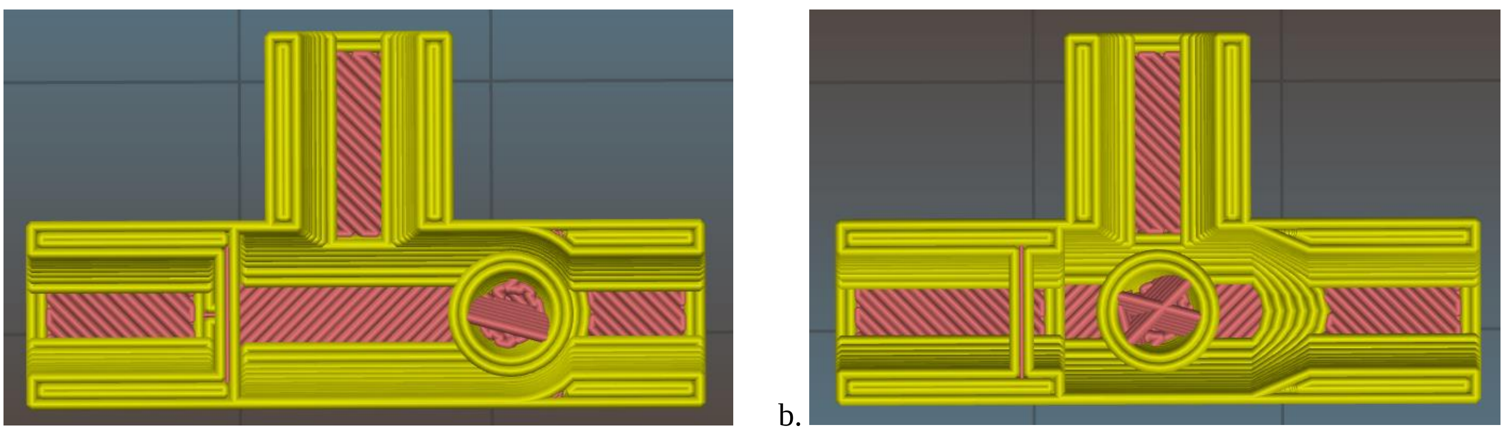


Figure 7. Cross section comparison of different valve designs. Figure a. is the original design with spherical cavity. Figure b. is updated design with conical cavity.

Figure 7. gives a bird's eye view into the inner workings of the valves. The valve in a. is the original design which led to considerable leaks when tested. Figure b. is the most recent version of the valve design. When qualitatively tested using the same method as for the original design, the valve with the conical cavity resulted in remarkably less leakage. This may be because the ball has more of a chance to get wedged into creating a good seal with the tapered end of the channel.

A simple method of quantifying the efficacy of the two different designs was devised, but unfortunately no data was obtained from the early qualitative tests. The T-shaped channels were connected to tubing as previously described, but the delivery of the water was controlled using the syringe pump component of OMIS. Unlike before, a third piece of tubing was also connected to port 3. The ends of the tubes connected to ports 2 and 3 were placed in separate 5 mL graduated cylinders. The flow rate of water delivered to the channel was set using Arduino. The amounts of water which flowed through ports 2 and 3 were compared once the syringe had delivered all the water. However, the flow rates achievable by OMIS are on the scale of µL/min, not nearly sufficient enough to supply the force needed to move the ball into the position to block the passage of liquids. Therefore no reliable data could be collected from these preliminary quantitative tests.

CONCLUSION

The initial goal of this project was to design a reciprocating pump such as one used for HPLC and print the pump monolithically. Due to unforeseen limitations from COVID-19, only the ball check valve component of the reciprocating pump was designed. The coded software OpenSCAD was used to design the valve. It was proven that a ball-in-socket type device could be made by simply printing a sphere on top of a support pillar in an otherwise hollow cavity. The ball-in-socket design was then implemented into a check valve and, later, into a T-shaped channel to test the viability of the valve. After several rounds of qualitative tests, it appears that a cone-shaped cavity is optimal for use in the valve, but the efficacy of its performance over that of a sphere-shaped cavity was not quantified. Quantification of this property using the method described is the first step that must be taken in future directions. Thereafter, the reciprocating piston can be designed. This can be in the form of a 3D printed piston that operates using a cranking mechanism or perhaps in the form of a syringe connected to the valve device with a Luer lock. After the piston is designed it can be implemented into the device, along with two ball check valves, to create a prototype of the reciprocating pump. Once the pump is created, a method of testing the pressure achieved by the pump will be devised.

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