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Drink Up: A Study of the Food-Safe Quality of Ceramics Glazes with the Addition of Rutile

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Drink Up: A Study of the Food-Safe Quality of Ceramics Glazes with the Addition of Rutile

An Honors Thesis submitted in partial fulfillment of the requirements for Honors in the *Betty Foy Sanders Department of Art.*

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Under the mentorship of *Professor Kimberly Riner*

ABSTRACT

Food-safe ceramic glazes can be altered with additives and become harmful to the user of the ceramic ware. Rutile is a frequently used material added to glazes to create variegation in glazes, but it is commonly known to cause defects in the glaze that can be unsafe for food. This experiment is conducted to determine if rutile can be added to food-safe glazes and still retain their food-safe status. A food-safe glaze is a shiny, thin coating that does not leach chemicals or has an excess of colorants; common food-safe glazes are white liners and clear glazes as they have no additives.

To test that food-safe glazes cannot be food safe with larger additions of rutile, an experiment testing seven different percentages of rutile in four glazes, on five commonly used clay bodies was conducted. 140 cups and test tiles were glazed in the 140 variations and put through seven separate tests to verify their food-safe status. The results showed that sixty-seven out of the 140 variations are food-safe, the majority, twenty-six of the sixty-seven, from John Britt's Spearmint glaze; conversely, John Britt's Licorice only had eight variations that were food-safe, five of which were the control cups with no rutile added.

The results suggest that rutile can be added to ceramic glazes in small amounts, depending on the clay body being used. With these results, rutile is a viable option to add these specific ceramic glazes and still be food-safe.

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Although the dating of ceramics is not specifically known, clay containers have been dated to between 20,000 to 12,000 BCE. The containers were used to process and store foods by hunter-gatherers.[1] Around 3,600 BCE, glass, a key component of ceramic glaze or the shiny, hard coating on clay vessels, was used as sharp cutting tools from naturally occurring sources like obsidian. Glass was not man-made until between 3,000-2,000 BCE in Egypt and Mesopotamia.[2] Glazed pottery has been dated to around 2,500 BCE from Ancient Egypt in the form of self-glazing clay called Egyptian paste. This clay contained sodium salts; this covered the surface of the clay and melts to create the glazed surface.[3] Glazes were used to seal the vessels to keep the moisture and bacteria out.[4]

Glaze Makeup

Glazes are made of materials that fall into four main categories, of which the first three are necessary: glass formers, fluxes, refractories, and additives. Glass formers make glaze hard, shiny, and durable. Glass formers seal the surface of the work and the most common material for this is called silica, or flint. Glass formers cannot be used by themselves because they have very high melting points.[5] The high melting points are incompatible with both clay bodies. The addition of fluxes solves the issue of high melting points.

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[1] O. E. Craig et al. "Earliest Evidence for The Use of Pottery," Nature 496, no. 7445 (2013): 351. doi:10.1038/nature12109.
[2] Alan Macfarlane and Gerry Martin, "Glass in the West – from Mesopotamia to Venice" in *Glass: A World History*, ed. Alan Macfarlane and Gerry Martin (Chicago: University of Chicago Press, 2002), 10.
[3] Mark Burleson, *The ceramic glaze handbook: Materials, techniques, formulas*. (Asheville: Lark Books, 2003), 8.
[4] Mitchell Beazley. "Materials and Techniques" in *Miller's Antiques Encyclopedia*, ed. Judith Miller (London: Penguin Press, 2003).
[5] Gaurav Madan, *S. Chands Success Guide (Q&A) Inorganic Chemistry*. (India: S. Chand Limited, 2005), 694.

Fluxes lower the melting points of glass formers so they can melt at temperatures the clay body can also withstand. Common fluxes are feldspars, such as potash feldspar and soda feldspar.[6] A glaze that contains too much flux will run off the clay body and a glaze with too little flux will not move and become a solid surface, or it will become bumpy and be underfired. Additionally, if fluxes are underfired, they can be toxic.

Refractories are considered the glue that holds everything together. Refractories stiffen the glaze enough to keep it from running off the piece, and allows the glaze fuse to the clay. A common material that is used in glazes for this purpose is aluminum oxide; the refractory is usually added in the form of a little bit of clay or alumina hydrate. If refractories are absent from the glaze, it would fall right off the clay body.[7]

The final category of materials added to glazes falls under the category of additives. These can vary greatly in amounts and combinations. Additives can include stains, oxides, opacifiers, and other materials. Stains are a blend of oxides and metals that are ground fine and are stable; stains are consistent and repeatable. Oxides are compounds of metallic or metalloid elements with oxygen and have high melting points.[8] These can help in each of the three essential categories, but certain oxides bring color to a glaze. Chrome oxide is bright green and it can turn a glaze green, or red when mixed with other oxides, such as tin oxide. Opacifiers are used to create an opaque quality to the glaze; The clay body cannot be seen through the layer of glaze. Sometimes you get opaque, white base glaze because

[6] Madan, *Inorganic Chemistry*, 694.
[7] Madan, *Inorganic Chemistry*, 694.
[8] Steven. S. Zumdahl, "Oxide." *Encyclopedia Britannica*, ed. Steven Zumdahl. 2018. https://www.britannica.com/science/oxide

some essential materials have opacifiers in them, such as whiting; whiting is a flux but also opacifies the glaze.

The first three categories of materials are the essential components of glaze. A glass former, flux, and refractory together would make a base glaze that is clear and transparent.[9] Some percentages of materials would need to be adjusted to get results suited towards their purpose, but these will make a working glaze. Once a base glaze is made and works as intended, the final group of materials can be added.

Glaze Issues

Adding a glass former, flux, and refractory together does not guarantee the glaze will work. Most materials are not solely made of just one category, but a mix of two or more categories. If a glaze is not balanced, three common issues can occur. The glaze can peel off the clay piece; this occurs from not enough refractories in the glaze.[10] The glaze can be porous from not enough glass formers or not enough fluxes; some people like the matte or bumpy look of the glaze, but they are not balanced glazes. Lastly, the glaze can crawl from too many fluxes or not enough refractories; the glaze pools off and at the bottom of the piece and peels off the body in thinly glazed sections.[11] All of these issues mean it is highly likely the glaze is not food-safe, even if appears food safe.

A balanced ceramic glaze is beneficial for storing and preservation of food and water. A balanced, food-safe glaze seals the vessel in a strong, durable coating. The glaze keeps

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[9] Gabriel Kline, *Amazing Glaze: Techniques, Recipes, Finishing, And Firing*. (Minneapolis: Voyageur Press, 2018), 21.
[10] Madan, *Inorganic Chemistry*, 694.
[11] William T. Brodie "Glaze Defects in Sanitary ware Plants," *Ceramic Engineering and Science Proceedings* 16, no. 3 (1995): 21

bacteria, mold, moisture, fungus, and air out. This is ideal to store foods and sensitive materials without the worry of contamination. Dinnerware and other food and drink vessels also benefit from food-safe glazes. Food particles cannot get trapped and grow mold and bacteria, and toxins do not leach from the work and harm the user in balanced, food-safe glazes. Continuing, many potters use these glazes to preserve their art. Unlike paints, papers, and waxes, food-safe glazes are archival.

A glaze must have specific qualities to be considered food safe. The glaze must be glossy and not matte. There cannot be any toxic base materials, excessive colorants, nor excessive stains. If a colorant, such as cobalt carbonate, is added at a 5% dry weight when 1% would give the same results, the excess can leach out of the glaze. Mason Stains can leach, or seep out, at higher temperatures so they would need to be thoroughly tested.

Continuing, the glaze cannot have physical defects, such as cracks and pores, nor can it have crystalizing. Crystalized areas in glazes are pockets of titanium frit that burst and spread across the clay body.[12] These points are weak and can collect bacteria in rough areas. Brightly colored or matte glazes are also not food safe because they potentially leach materials harmful to the user.

Common food-safe glazes do not contain any additives to be confident they are food-safe. Clears, white liners, and transparent overglazes (a low-temperature glaze that is used as a second glaze layer) are all commonly thought of as food safe, but thorough testing should be used to confirm. Non-food safe glazes can be detrimental to the user of the

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[12] Do Quang Minh et al., "The Novel Crystalline Glaze for Decoration of Ceramic Pottery." *Materials Science Forum* 987, (April 2020): 165. 10.4028/www.scientific.net/MSF.987.165.

ceramic piece. A glaze can shed glass shards, hoard bacteria, and leach toxic heavy metals and these can come from several issues.

Common Defects in Glazes

The most noticeable issue that can occur are visual defects: pinholes, blistering, crawling, and cutlery marking. Pinholes are small holes that appear in a glaze; these holes go directly to the clay body and can collect food particles that are difficult to clean.[13] The pinholes can occur when a material in the glaze gives off gases in the firing and does not close. A thick application of glaze or a fast firing can also encourage pinholes.[14]

Figure 1. Example of pinholing (Photograph by Tony Hansen. “Pinholing” *Digital Fire*, 2018.)[15]

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[13] Cyril Harris, "Pinhole" In *Dictionary of Architecture and Construction*, ed. Cyril M. Harris (New York: McGraw-Hill, 2006), 728.
[14] Kline, *Amazing Glaze*, 146
[15] Tony Hansen, “Pinholing” *Digital Fire*, accessed August 25, 2021, https://digitalfire.com/trouble/pinholing

Blistering is similar to pinholes as it occurs from off-gassing in the firing. Blisters appear similar to a cluster of bubbles or foam. They occur from gases escaping the glaze when heated, however, there is too much surface tension and holds the gas.[16] They can also occur from a glaze that is not overly melted and is too thick to release the gases.[17]

Figure 2. Example of blistering (Photograph by Tony Hansen. "Glaze Blisters" *Digital Fire*, 2018.)[18]

Additionally, crawling is another visual defect in the glaze. The glaze separates from the clay body in sections or it shrinks away from itself, both creating bare patches.[19] It occurs when there are issues in the application of the glaze or too much raw clay in the glaze. Clay is used to add glass formers and refractories to the glaze, but it can be overpowering and throw the balance off.

[16] Hansen, "Glaze Blisters"
[17] Dave Finkelnburg, "Blistering." *Ceramics Monthly* 62, no. 10 (November 2014): 65.
[18] Hansen, "Glaze Blisters"
[19] Kline, *Amazing Glaze*, 148

Figure 3. Example of crawling
(Photograph by Tony Hansen. “Crawling”
Digital Fire, 2018.)[20]

Cutlery marking is a result of a glaze being porous and soft from an imbalance in the glaze or certain additives increasing the chances of cutlery marking. The glaze is commonly matte or cloudy.[21] When a metal utensil is scratched into the surface, a dark mark appears on the glaze. If the mark does not wipe off, the glaze is soft and can trap bacteria in its pores. This comes from a lack of enough glass formers in the glaze.

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[20] Hansen, “Crawling”
[21] Zeke C. Seedorff, Richard C. Patterson, and Heinz J. Pangels. "Testing for metal marking resistance." *Materials and Equipment-Whitewares* 146 (2009): 196.

Figure 4. Example of cutlery marking (Photograph by Tony Hansen "Cutlery Marking" *Digital Fire*, 2018.)[22]

Another food-safety concern comes from the relationship between the glaze and the clay body. Crazing and shivering are more difficult to see on freshly fired work- they show up months, even years later.[23] Both crazing and shivering leave areas for bacteria and mold to grow in the piece. The issues stem from both glaze and clay body slightly shrinking and expanding during fluctuations in temperatures as they shrink and expand at different rates.

Crazing looks cracked and covered with tiny squares. The glaze can also be compared to a broken shell of a hard-boiled egg or a shattered car window. This is from the glaze shrinking away from the clay body or the clay body expanding more than the glaze.[24] As food fills the crevices, the lines become more prominent.

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[22] Hansen, "Cutlery Marking"
[23] Jeff Zamek, "A Simple Glaze: Jeff Zamek Demystifies the Chemistry of Glaze," *Ceramics Technical*, no. 39 (2014): 70.
[24] Kline, *Amazing Glaze,* 149

Figure 5. Example of crazing (Photograph by Tony Hansen. "Crazing" *Digital Fire,* 2018.)[25]

Shivering is the opposite of crazing. It looks as if the glaze is flaking off the clay body and can leave bare patches.[26] If a finger is run over it, it can sound like the crunching of dry leaves and can feel sharp. The glaze does not shrink as much as the clay body shrinks in cold temperatures, leaving a gap, or the glaze has expanded more than the clay body under heat.

[25] Hansen, "Crazing"
[26] Kline, *Amazing Glaze,* 149

Figure 6. Example of shivering (Photograph by Tony Hansen. “Shivering” *Digital Fire,* 2018.)[27]

Lastly, leaching is the unseen danger of unbalanced glazes. Harmful chemicals found in the glaze can get into foods and drinks, especially from very acidic or alkaline foods. This is very important and needs to be taken seriously, especially if using toxic materials. This can come from the glaze not fully melting, an excess of toxic materials, or a lack of glass formers.[28]

Testing

Each of these defects is tested extensively for the safety of consumers and the reliability of the glaze. If a glaze is reliable and food safe, the glaze can be reused and does not need to be tested for each piece. A potter should not sell their work as food safe if they have not

[27] Hansen, “Shivering”

[28] Taiwo A. Aderemi, Adeniyi A. Adenuga, John A. O. Oyekunle, and Aderemi O. Ogunfowokan. “High Level Leaching of Heavy Metals from Colorful Ceramic Foodwares: A Potential Risk to Human.” *Environmental Science & Pollution Research* 24, no. 20 (July 2017): 17116. https://doi.org/10.1007/s11356-017-9385-7.

tested their glazes; the creator could potentially harm the user of their work if the potter does not test their glazes.

Several tests are performed to test for issues within the glaze. The tests are performed in an order of visual inspection, cutlery test, boiling water: ice water test, lemon slice test, glaze leaching test, soda ash test, and lastly the microwave test. The visual inspection is simply looking at the piece to see for defects: pinholing, blistering, or crawling. If the piece does not pass the visual inspection, it is deemed unsafe for food and does not need further testing.

The cutlery test tests the softness and porosity of the glaze. A grey smudge may appear and, after washing the piece, if the grey smudge does not disappear, this means the glaze is unbalanced and can trap bacteria after many uses. The structure of the glaze has a microscopic rough surface that is hard to see with the naked eye.[29] The glaze would not be food-safe.

The boiling water: ice water test tests extreme temperatures and the shivering or crazing that may occur. The ceramic piece is boiled for three minutes then plunged into ice water for three minutes. This is repeated three more times at each temperature. It shocks the piece into rapidly shrinking and expanding. Ink is brushed on and rapidly wiped off to mimic food being trapped in any crazing or shivering. Crazing shows lines or squares but shivering shows splotches of ink that can't be washed off- it is trapped under the glaze.

The lemon slice test is one of three tests for leaching; this one is for acidity. A lemon slice is left on the glazed portion of the pieces for ten days and is covered to prevent the

[29] Seedorff, Patterson, and Pangels, "Metal Marking Resistance," 196.

lemon slice from drying out.[30] A weighted piece is put on the lemon slices to keep the most contact of the lemon and glaze. If there is discoloration from under the lemon, there is an issue in the glaze and is leaching a material.

The glaze leaching test is another test for acidity. The piece is filled halfway with vinegar. It is then left alone for ten days and covered to prevent evaporation. Any discoloration to the vinegar or the piece itself is a sign of leaching- this includes lightening, darkening, a change in the texture of the glaze, or cloudiness.

The soda ash test is performed to test alkaline materials on the glaze. The process begins by boiling a single glazed piece in fifty grams of soda ash combined with one liter of water for six hours. A half-liter of water is added every half hour. If there is a color change or clouding of the soda ash mixture or glazed piece, there is an issue in the leaching of the glaze with alkaline materials.

Lastly, the microwave test identifies high amounts of iron and any missed or unseen issues that may occur. These issues may be hard to notice until years of wear and tear. The issues include high iron content, under-firing, porosity issues, tiny pinholes, or slight crazing hidden by the variegation in the glaze. A small amount of water is poured into the bottom of the piece and microwaved for thirty seconds. If the glazed piece is hotter than the water, there is an issue with the piece that can be from the clay, glaze, or a combination of both.[31]

[30] Kline, *Amazing Glaze*, 150
[31]Kline, *Amazing Glaze*, 151

Rutile

A glaze needs to be tested when we add an ingredient into an already food-safe glaze. The material could affect the balance of glass formers, refractories, and fluxes; this could then cause leaching or soften the structure of the glaze and make it porous. Rutile is added to food-safe glazes and tested to see if the addition caused the glaze to become unsafe.

Rutile is a mixture of over 85 percent titanium dioxide with around 15 percent impurities; if the percentage of impurities is higher, it is called ilmenite, not rutile.[32] Rutile is commonly referred to as weld rod titanate outside of ceramics and is used in welding. In pottery, rutile is known to cause variegation in glazes. Variegation is not a homogeneous solid color, but a mix of crystal growth, speckles, phase separation, layering, and opacity variations; it is highly prized for its uniqueness and one-of-a-kind quality. Rutile is also mixed into the clay to make speckles within the clay that can shine through and interact with a glaze applied on top. There are different sizes of rutile and each is used differently. The three types of rutile are granular, dark, and light.

Granular rutile is primarily used for speckles in clay bodies as it is very heavy from its large particle size. Granular rutile is ideal for clay because of its size and weight. The weight does not allow it to suspend in glaze easily, but some potters use it in their glazes. The issue with using granular rutile in glazes is the large particle size in glazes can cause larger pinholes and blistering as more gases are burning out. It is also difficult to apply as the glaze needs to be constantly stirred to keep the granular rutile suspended.

[32] Ryan Coppage, “Rutile,” *Ceramics Monthly*, October, 2017

Dark rutile is raw, uncalcined rutile and has a smaller particle size than granular rutile. The rutile has not been calcined and has a dark or blue cast to it. Calcination is a purification process that oxidizes a portion of the material at high temperatures.[33] Dark rutile is used in glazes, but it is not as commonly used as light rutile.

Light rutile is a fine particle, calcined rutile. It is made of 90% titanium dioxide with only 10% impurities. When rutile is calcined, it turns to a light tan color and a portion of the impurities are burned out.[34] Light rutile is mainly used for glazes as its particle size is ground to 325 mesh- fine enough to suspend in glazes.

Rutile can opacify glazes in high quantities, around 6-7% of the dry material weight of the glaze.[35] At this high of rutile, it can weaken the glaze and become porous. If the glaze opacifies, the glaze does not have variegation. However, rutile releases gases as it is heated, so pinholes, blistering, and crawling are common.

The Experiment

The Controls

All of the work in this experiment was first fired, or heated, to 1000 °C or 1828 °F. At peak temperature, the clay vitrifies and becomes ceramic; at this stage, the work has been bisqued. This means that the clay has hardened and the porous quality of the clay is minimized. The temperature is referred to as cone 06.

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[33] B. Rand, “Calcination”. in *Concise Encyclopedia of Advanced Ceramic Materials*, ed. Richard J. Brook (New York: Pergamon Press, 1991), 49.
[34] Coppage, “Rutile.”
[35] Richard Eppler, "Controlling Glaze Surface Effects," *American Ceramic Society* 81, no. 9 (2002): 27.

Cone 06 is a level on the Pyrometric Cone Chart. A “cone” refers to the temperature of a small pyramid, called a pyrometric cone, which softens and deforms. Each “cone” softens at a different temperature and the chart ranges from cone 022 (586 °C or 1087 °F) to cone 14 (1365 °C or 2489 °F). Pyrometric cones and the Pyrometric Cone Chart are used to track and monitor temperatures in the kilns to ensure consistency and fully fired kiln loads.

The glaze is applied after the first firing and then needs to be re-fired to cone 6 temperatures. The clays and glazes used need to reach cone 6, 1220 °C or 2232 °F- a midrange temperature that is commonly used by potters.

In addition to cone 6 temperatures, all work is being fired in electric, oxidation kilns. Electric kilns use electricity, not natural gas to reach the needed high temperatures. The electric kiln is an oxidation atmosphere, meaning there is oxygen in the firing.[36] Electric kilns allow for more control over the temperatures needed for both the first and second firings. The work is fired in a single batch, both for the bisque firing and the second, glaze firing.

Four cone 6 glaze recipes are used: John Britt’s Licorice[37], Odyssey Clay Works’ Fat Cat Red[38], Ron Roy’s and John Hesselberth’s Ol’ Blue[39], and John Britt’s Spearmint[40]. Glazes are named after the potter who developed the glaze. Many potters develop multiple glazes and share them with the pottery community. For example, John Britt and his glazes are well known in the ceramics community.

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[36] Kline, *Amazing Glaze*, 25
[37] See Appendix 1
[38] See Appendix 2
[39] See Appendix 3
[40] See Appendix 4

John Britt's Licorice is a rich black glaze that breaks brown on thinner areas of glaze. The glaze does not run, or flow down, the side of the vessel. It is slightly translucent but the clay body only affects the glaze in its thinner areas.

Additionally, Odyssey Clay Works' Fat Cat Red is a warm red glaze that does not run. It is translucent and in its thinner areas, it appears orange. Ron Roy's and John Hesselberth's Ol' Blue is a true cobalt blue that does not run down the vessel. The clay body greatly affects the look of the glaze.

John Britt's Spearmint is the last glaze in this experiment. It is a mid-tone green and can be runny if layered thick. The glaze is translucent and the clay body affects the look of the glaze greatly.

A clear or white glaze was not used as it is hard to see any variegation in these glazes. In addition, many variegated glazes are darker colors, such as black and navy. These four glazes were chosen as they are commonly used by potters and are published recipes. However, a published and common glaze does not mean it is safe for vessels used for food and drink.

Different clays can affect the look of the glaze as well as the compatibility between the glaze and clay body. Five common types of stoneware clays are used to test if the glaze is food safe and if variegation is more prominent on a specific clay type. Stoneware clays are high-fired clays that, when fully fired, become non-porous and durable. They are commonly used for dinnerware and can be used in the microwave and oven because it distributes heat more evenly than other types of pottery. The clays are cone 6 recipes and the five variations are porcelain[41], white[42], speckled[43], brown[44], and reclaim.

The brown clay has an addition of 3 percent red iron oxide to get its darker hue. The clay is a smooth, homogeneous warm brown. The bisqued piece looks pink but darkens in the cone 6 firings. The speckled clay is slightly tanned with 1 percent red iron oxide and has 1 percent granular ilmenite added for the speckles.

The reclaim clay is made up of scraps of mixed clays used by a potter or ceramic studio. This is common for potters to reuse the clay scraps from other projects. The reclaim clay used is from the Georgia Southern University Armstrong campus ceramic studio. This mix is heavy in grog from different types of clay used in the studio. It has a pale beige tone when fired from darker clays mixed together. White clay is a commonly used clay. It has a slightly off-white tone and has no colorants added to it.

Porcelain is the final clay used to test the glazes. Porcelain is usually a translucent, white clay body that is non-porous. It is delicate and has very fine particles, but it is hard to work with. It can crack easily from stress spots and thin areas because it has small workability and low plasticity; the more the clay is manipulated, the more issues occur. Porcelain is prized for its pure white color and slight translucency when fired to full temperature. There are two main types of porcelain: cone 6 and cone 10, a higher temperature clay. Cone 6 porcelain was used in this experiment.

[41] See Appendix 5
[42] See Appendix 6
[43] See Appendix 7
[44] See Appendix 8

All materials for the glazes and clay are sourced from the same batches. One large order was made from The Ceramic Shop for light rutile, red iron oxide, wollastonite, Gerstley borate, and granular ilmenite. A separate order was made from Fort Pottery for large amounts of silica, OM4 ball clay, Nepheline Syenite, Custer Feldspar, frit 3195, tin oxide, and Edgar plastic kaolin. Any small amounts of materials were from the Armstrong ceramic studio, purchased in large quantities.

To keep consistency between tests, the shape of the vessel and test tile are kept identical. The vessel is a faceted cup- a common form made by potters and is similar to multiple types of forms made. It also has several characteristics used in ceramics: vertical and horizontal surfaces, thin edges, and texture - both smooth and raised. The facets show how the glaze would deal with a textured, or raised surface, and the high point of the rim shows the thinness of glaze- and if it affects the glaze issues. The same cup shape allows for comparison between these areas. This form can also be filled for the glaze leaching test.

The test tiles are used by potters to test glazes and see how the glazes will look on a form. The test tiles were made to pair with the cups- as it is a check on the glaze to see if an issue in the glaze is from the whole batch or just an individual issue.

To keep everything identical, the cups and test tiles were slip-cast. Slip casting keeps everything identical by using a mold. This keeps the thickness and shape of the cup similar between the clays to keep inconsistencies at a minimum.

Figure 7. Unfired cups waiting to be loaded into the kiln.

Slip Casting

Slip casting is the process of pouring liquid clay into a plaster mold to firm up and produce a hollow vessel. There is a very small margin of error from the molds, but there can be a higher risk of damaged vessels when they are released from the molds. The unfired pieces break easily because they are fragile and thin. Around fifty cups of each clay were made, and seventy cups were made for porcelain because of their difficult nature. Extras were made in case of breakage, inconsistencies, and testing for errors.

Figure 8. Bisqued cups stacked after coming out of the first kiln firing.

To begin slip casting, the process begins in making the plaster molds. As always, a respirator with N100 filters is worn to keep the plaster from entering the body and collecting in the lungs and the process is done outside or in a well-ventilated room. To make the molds, 125 pounds of plaster, water, Murphy's oil soap, five feet of six-inch diameter garden irrigation pipe, eight worm drive hose clamps, a tube of silicone, clay, a drill with a mixing attachment, and eight of the vessels that are being molded are needed; the vessel chosen was a glass, faceted cup.

Figure 9. Glass cup used as the
vessel for the plaster molds.

The garden irrigation pipe is cut into eight equal pieces and a vertical line is cut into the pipe for easy release.

Figure 10. A section of the
garden irrigation pipe cut
vertically.

The cup form was secured to the tabletop by its opening (the rim of the cup against the table) with the silicone; this creates a tight, waterproof seal. The pipe is held together with a worm drive hose clamps and secured around the cup so there is equal space between the cup and pipe with clay on the outside and silicone on the inside. The clay and silicone act as seals so the plaster cannot escape.

Figures 11-12. The glass cups and garden irrigation pipes secured to the table with clay and silicone.

Everything was brushed with the oil soap- this stops the plaster from sticking and makes the glass cup slide out easier. The plaster was mixed up with the drill attachment in a ratio of two parts plaster to one part room temp water- this leads to a good pouring consistency. Around twenty-five pounds of plaster were used for each round of eight molds.

Figure 13. Plaster is being added to a five-gallon bucket.

The plaster was poured into each mold and the sides of the molds are tapped to release air bubbles. If the plaster was thickening, a paintbrush was used to brush the sides of the cup to release the air bubbles.

The molds set for twenty-four hours and the pipe and cup are released. The process is repeated four more times to make thirty-two molds. The molds were dried for four weeks before they can be filled with slip.

Slip

Slip is clay that has been liquified with a mixture of water and deflocculant. A deflocculant is a chemical that, when added to a substance, allows the substance to become more fluid without the addition of a liquid; it thins a substance without diluting it. The following is needed to make slip: all dry materials for the slip recipe, a five-gallon bucket, water, a drill with a mixing attachment, scales that weigh to the hundredth of a gram, coarse 12 mesh sieve, fine 80 mesh sieve, and Darvan 7 are needed.

Before beginning, a respirator with N100 filters needs to be worn because many of the materials in clay are harmful to the lungs and body in their dry form and everything is mixed in a well-ventilated room. When starting, all dry materials are calculated for 20,000-gram batches of dry materials for each five-gallon bucket; two 20,000 grams of dry slip are needed for each clay. For both slip and glaze recipes, the materials are given in percentages and need to be tailored for the amount needed. If a recipe needs 19 percent silica for a 20,000-gram batch, 3,800 grams of silica would need to be added to the clay recipe.

After everything is calculated, 5,000 grams of water is added to each of the five-gallon buckets. This prevents the dry materials from flying into the air as much. The dry materials are added after each is carefully weighed. The buckets are then mixed up and extra, weighed out water is added until the mixture is thick.

Figure 14. Slip materials mixed with
a small amount of water- not sieved.

The mixture is tested for its specific gravity. Specific gravity is the ratio of a substance and reference material (this experiment uses water) in a related quantity, such as volume.[45] Slip used for casting should have a specific gravity between 1.75-1.85 when compared to water. If the specific gravity is between this range, weighed out Darvan 7 is added gradually to deflocculate the mix, making it smooth and similar to a cream-like consistency.

Figure 15. Slip at a cream consistency- streaming smoothly off the whisk without clumps.

The slip then sits for twenty-four hours to hydrate any dry clumps in the mix. After twenty-four hours, the slip is sieved- first through the coarse sieve and then through a finer sieve. Sieving removes any unmixed sections and large materials. After sieving, the slip is ready to use. The slip is covered to avoid evaporation that would alter the specific gravity.

[45] Kline, *Amazing Glaze,* 151

The reclaim slip was made differently. The reclaim clay scraps were dried out and pulverized to a powder. 20,000 grams of the powdered mix was added into a five-gallon bucket with 5,000 grams of water. Again, the mixture is tested for its specific gravity. Darvan 7 was added and the mixture sits for twenty-four hours. It is then sieved several times. For the reclaim, this sieving process is critical as it removes excess and large grog that ends up in scrap clay. If not sieved thoroughly and multiple times, the grog can end up showing as lumps along the inside of the slip-casted pieces. After making the reclaim slip, it will not be used again as the grog could not be completely removed and left small lumps on the insides of the cups.

Making the Vessels

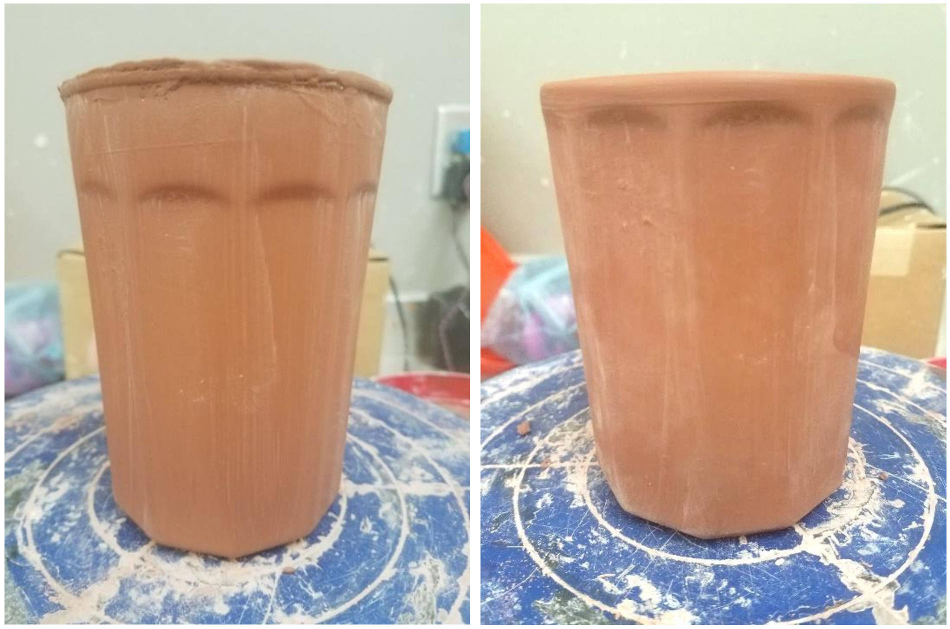
To pour the cups, both the dried-out molds and sieved slip is needed, as well as a whisk or stirring utensil. Before pouring the slip into the molds, the slip needs to be stirred to make sure the materials are completely incorporated and homogeneous. Around one quart of slip was poured to the top of the mold and let sit in the molds. The slip is left in the molds for one hour.

Figure 16. Plaster molds filled with slip.

As the slip comes into contact with the plaster, water is drawn out. This creates a layer of solid clay on the sides of the plaster. After an hour, around one-third inch of clay builds up on the sides. The remainder of the clay is poured out and the molds are left to dry for twenty-four hours until the pieces firm up.

Figure 17. Emptied plaster molds with a layer of slip drying along the inside form.

The clay becomes firmer and becomes closer to solid, wet clay. At this stage, the cup can be cleaned up. The top edges of the cups are trimmed and any excess on the sides or bottom of the cups is cleaned up.



` Figures 18-19. Before (Left) and after (Right) cleaning up the casted forms.

The cups are dried slowly for a week. This helps reduce the amount of breakage during the bisque firing. The cups were all fired together to rule out issues in the bisque firing of the kiln. All cups survived the bisque firing, with the exception of a portion of the porcelain cups. There were few that had crackling on the bottoms of the insides of the cups and there were some that cracked through the bottom of the cups.

The cups are labeled on the bottom so that the glaze cannot cover the labels and it is easier to see. They are labeled by Clay type/Glaze/Percent of Rutile with a key to shorten the writing. The key relates to each clay and each glaze. The percentage of rutile is labeled by the number 0-6. The keys for the clays and glazes are listed below:

Tables 1-2. Clay Body Key (Left) and Glaze Key (Right)

Clay Body Key	
R=	Red Iron Oxide (Brown)
RC=	Reclaim
S=	Speckle
W=	White
P=	Porcelain

Glaze Key	
L=	John Britt's Licorice
S=	John Britt's Spearmint
B=	Ron Roy and John Hesselberth's Ol' Blue
R=	Odyssey Clay Works' Fat Cat Red

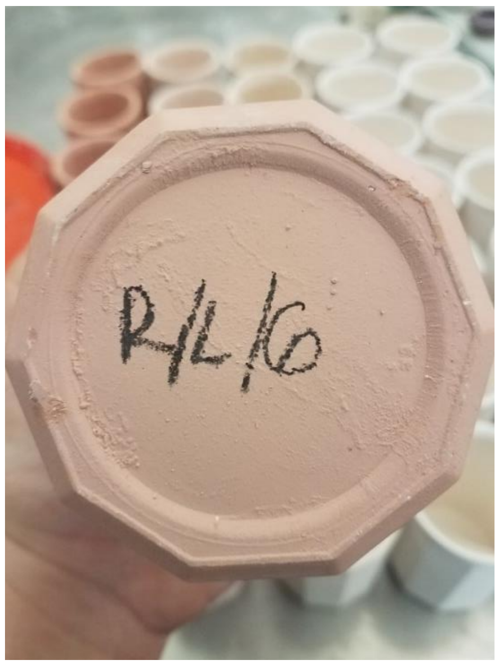


Figure 20. View of the labeling on the bottom of the cups.

The Glazes

Once the cups had been fired, the glazes were prepared. As with making the molds and slip, before mixing any dry materials, a respirator is used with N100 filters. Additionally, latex gloves are worn because some materials can absorb through the skin and harm the body. The glazes are mixed in 9,000-gram batches of dry materials. The light rutile was not added to the recipes yet as it would be added after the glazes are divided. To make the glazes, four five-gallon buckets, the dry glaze materials, light rutile, a scale weighing to the hundredth of a gram, a whisk, water, and a fine 80 mesh sieve. The glazes were calculated for 9,000-gram batches of dry materials; again, glaze recipes are given in percentages to make any size batch needed.

In making the glazes, one of the four buckets was filled with 5,000 grams of water to reduce the amount of dry material entering the air. The dry materials are weighed and added to the bucket. After all materials are added, the glaze is mixed and water is weighed and added until it reaches a light cream consistency, or a specific gravity between 1.35-1.45. The glaze then sits for twenty-four hours for the dry materials to absorb the water. The mixture is then sieved and extra water is added to fix the consistency, staying in the specific gravity range. This process is repeated for the three other glazes and then the glazes were ready for glazing the bisqued cups.

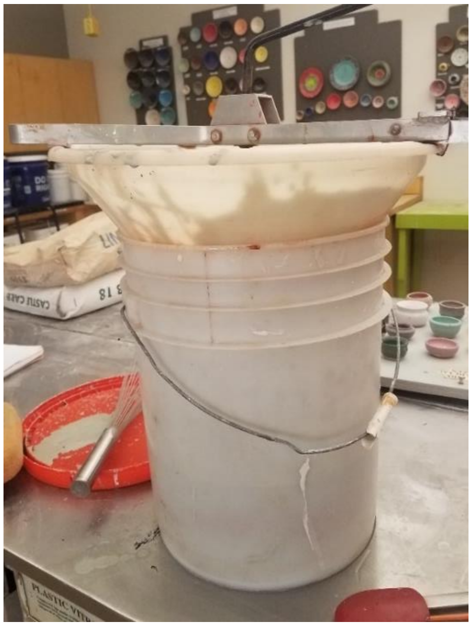


Figure 21. Sieve atop a five-gallon bucket.

Figures 22-23. Before (Left) and after (Right) of sieved glaze.

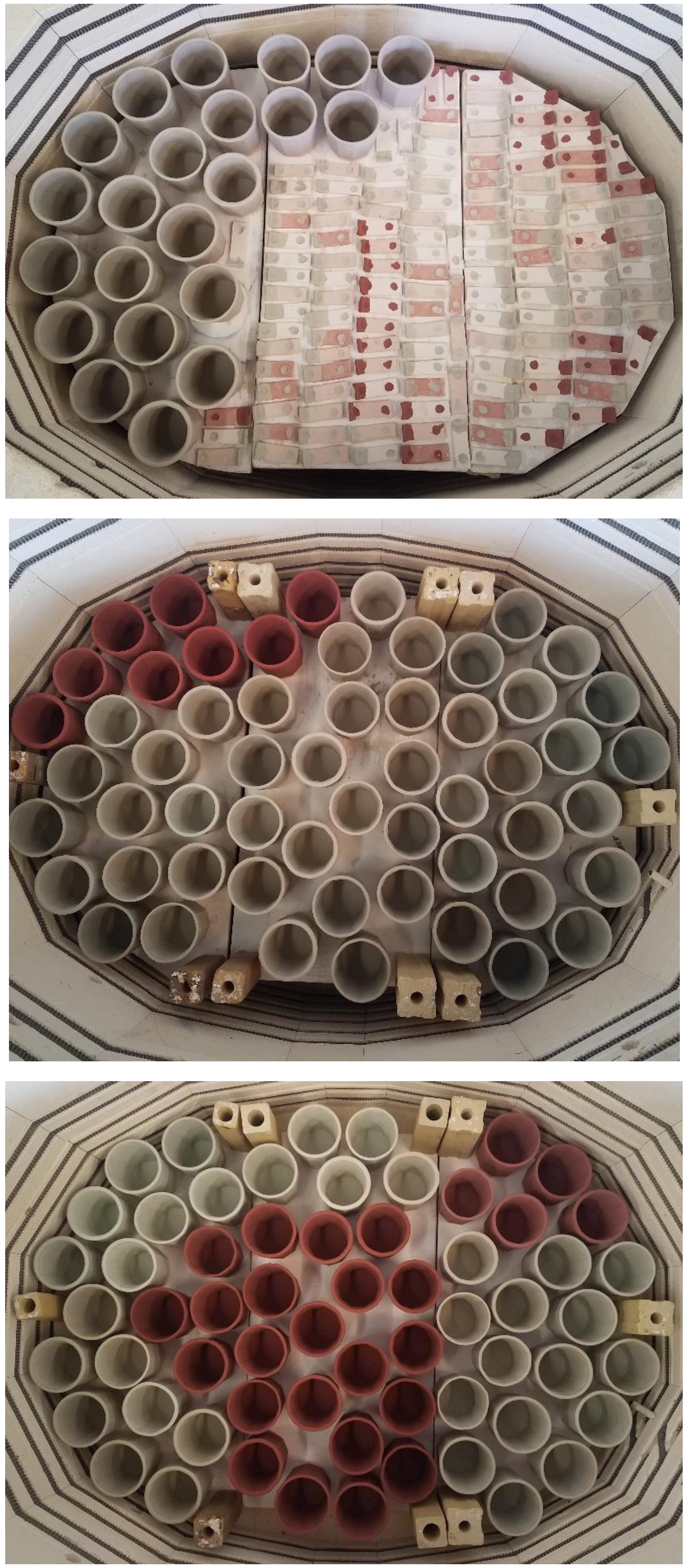
To glaze the cups, the glazes are separated into seven containers for each glaze. Rutile is added to the glazes by the dry material weight. There is a cup of each glaze on each clay body glaze without rutile as the control; the control cup should not have any issues and be food safe. An example would be to add 2 percent light rutile to a 9,000-gram dry batch of John Britt's Licorice glaze, 9,000/7= 1.285.71 grams and 2 percent of 1,285.71 grams is 25.71grams of light rutile added to the batch.

After all the glaze mixes were made, the glaze was applied to the cups. The cups were dipped in the glazes. This is a common method of application and it is easier to get the application as identical as possible. Other types of applications are more difficult to apply evenly. Brushing on the glaze is uneven as it is difficult to apply by hand with several coats that do not overlap; spraying is also is uneven and prone to thin spots because the glaze needs more water to be able to go through the sprayer and must be layered up similarly to brushing on the glazes.

When dipping the cups in the glazes, a ¾ inch area near the foot, or bottom, of the cup is left unglazed. This leaves room if a glaze has too much flux and drips down the piece. The dips are also timed- once the cup is quickly submerged, it is held in the glaze for one second and pulled out quickly.

Figure 24. Cup coming out of the glaze.

The cups are all dried together and fired together at a cone 6 temperature for the glaze to fully mature. They were fired in the same kiln as they were bisqued at and is again fired in an oxidation environment. The kiln was programmed to slowly reach peak temperature and then cool. The slow firing and cooling reduce the chance of pinholes and blistering from forming. The downside of a slow firing is that it can cause severe running if there is too much flux in the glazes.



Figures 25-27. The levels of the kiln before glaze firing.

Figures 28-30. The levels of the kiln after glaze firing.

Results

Physical Defects

After the cups were fired, the cups begin rounds of testing. All of the data was compiled into Excel sheets to follow trends and create graphs. The cups went through rounds of testing. If the cups do not pass a test, they do not move on to the next round. One hundred forty cups were tested with four glazes, on five clays, and with seven percentages of rutile each.

Figures 31-32. Odyssey Clay Works' Fat Cat Red and John Britt's Licorice (Left) and Ron Roy and John Hesselberth's Ol' Blue and John Britt's Licorice (Right) cups lined up in a grid formation.

Before firing the cups, there were a few predictions made after researching about rutile. Glazes made with 2-3 percent light rutile may be best for all clays. The Spearmint and Licorice glazes may have the most food-safe cups. All glazes at 6 percent light rutile will opacify and have no variegations. Crazing and blistering will be high in 4-6 percent because there may be too much flux and too many gases coming off from the light rutile.

The first test, the physical inspection was the major category that disqualified cups from being food safe. Out of the forty cups marked off for visual defects, ten cups had pinholing, six had crazing, five had crawled, and nineteen had blistering.

Figures 33-35. Examples of blistering (Left), pinholing (Center), and crawling (Right).

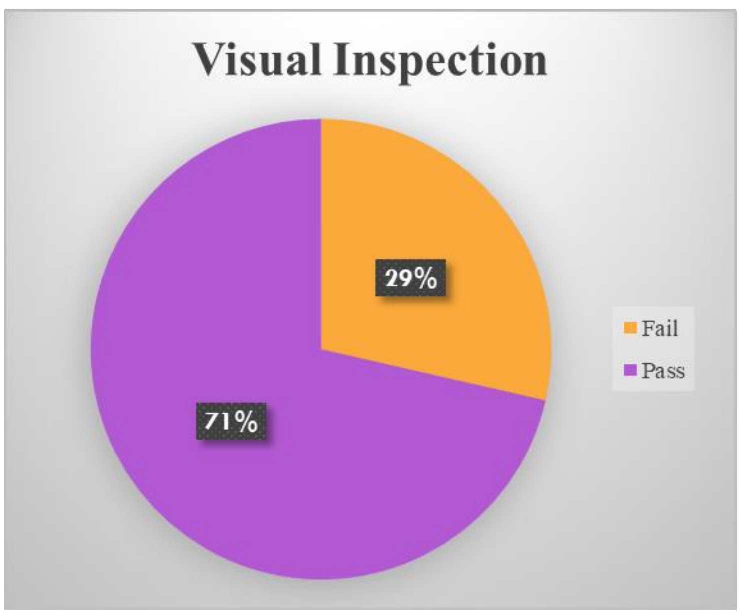


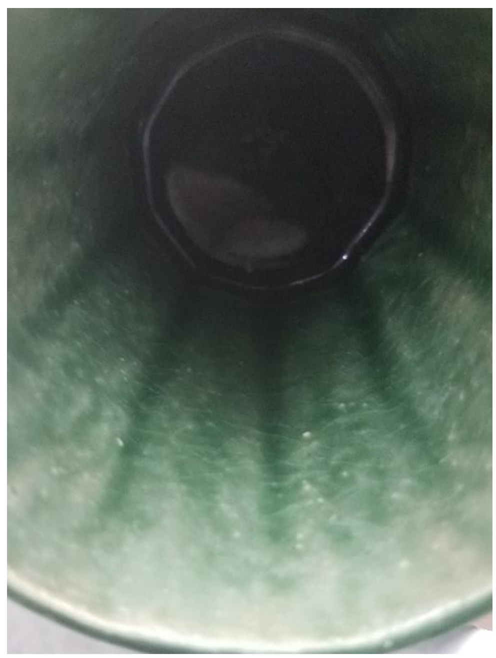
Figure 36. Graph of the pass/fail rate for the visual inspection

John Britt’s Licorice glaze had the most issues, especially from blistering and crawling- all nineteen blistered and five crawling cups were glazed in Licorice. Licorice was re-tested again and another test was done with a lower specific gravity of 1.25 (thinner). The tests had pinholing and had smaller blisters.

Figure 37. Pinholing and small blistering of the re-tested Licorice glaze.

The six visibly crazing cups were from John Britt’s Spearmint glaze on reclaim clay; the affected percentages were the 0-5 percent and the 6 percent was not affected. Of the ten cups that had pinholing, all were from Odyssey Clay Works’ Fat Cat Red. Pinholes appeared on the brown and speckled clays at 2-6 percent. After these cups were removed, one hundred cups remained.





Figures 38-40. Examples of crazing on Spearmint glaze. The top images show single hairline cracks and the bottom image shows severe crazing where it appears the glaze is rippling.

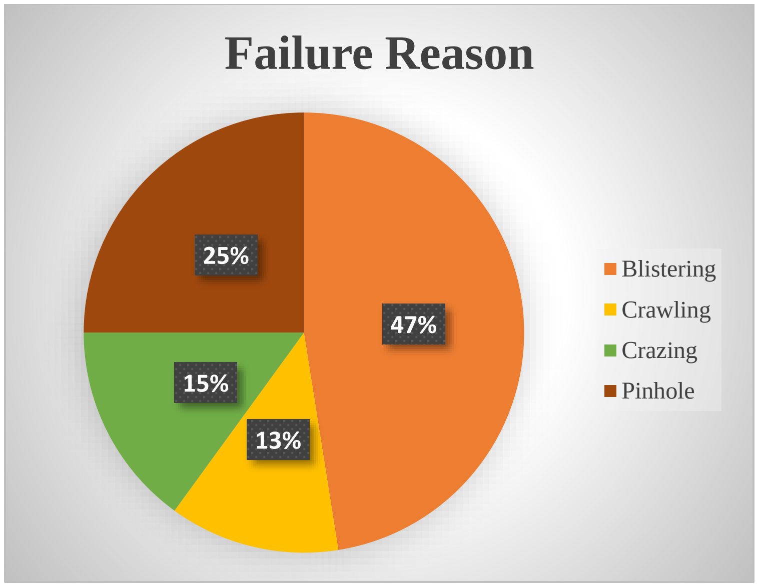


Figure 41. Graph of the reasons glazes failed the visual inspection.

Cutlery Marking

The next test was the cutlery marking test. This was the second biggest category of thirty-one cups removed. Of John Britt's Spearmint glaze, the 6 percent test on reclaim clay and the 5 percent and 6 percent tests on white clay had cutlery marking. Of Ron Roy's and John Hesselberth's Ol' Blue, the brown clay 5-6 percent, the speckled clay 3-6 percent, the reclaimed clay 2-6 percent, the white clay 4-6 percent, and the porcelain clay 5-6 percent all had cutlery marking. Of Odyssey Clay Works' Fat Cat Red, the white clay 3-6 percent and the porcelain clay 2-6 percent had issues with cutlery marking. Of John Britt's Licorice, the reclaimed clay had cutlery marking at 4-6 percent.

Figure 42. An “X” on a soft glaze showing the results of a cutlery marking test.

A pattern emerged that the cutlery marking was appearing at higher percentages of rutile. This could be from the glaze opacifying and becoming matte or porous. Of the one hundred cups, sixty-nine passed the second round of testing.

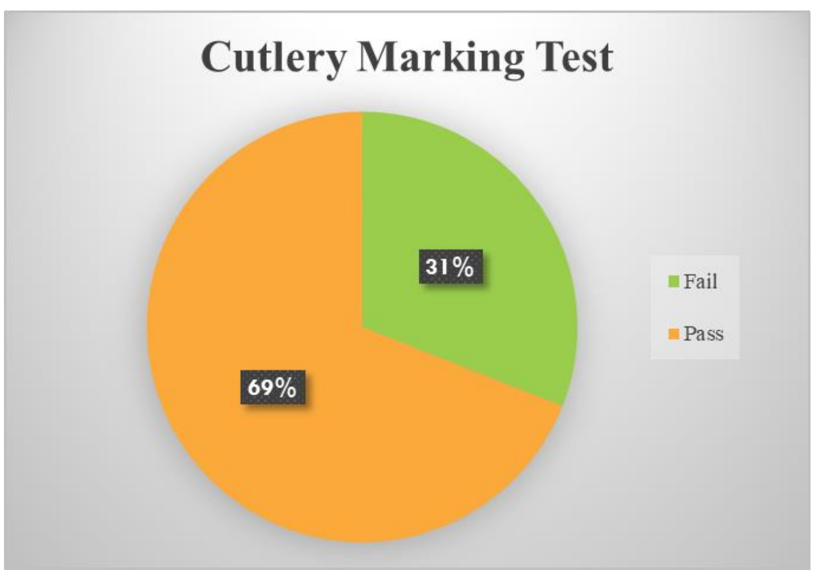
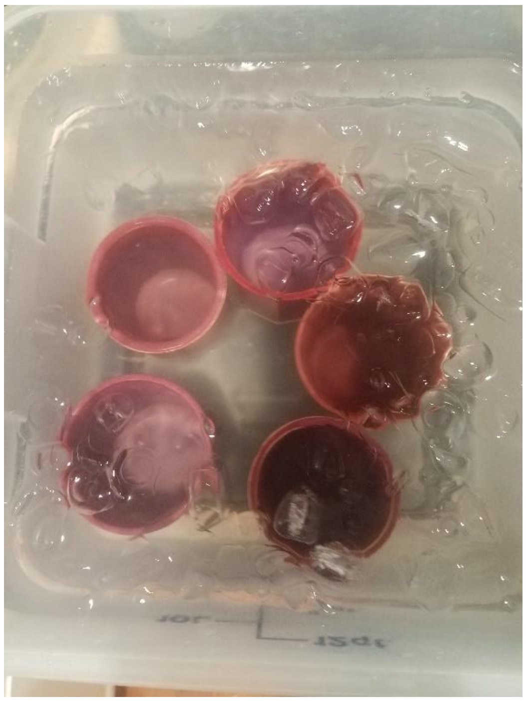


Figure 43. Graph of the pass/fail rate of the cutlery marking test

Boiling Water: Ice Water Test

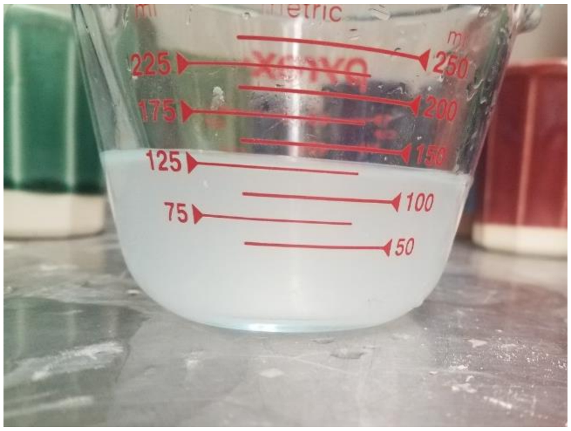
The third test, the Boiling Water: Ice Water test had no cups that had issues. The crazing seen in the first round were the only cups that had issues of either crazing or shivering.



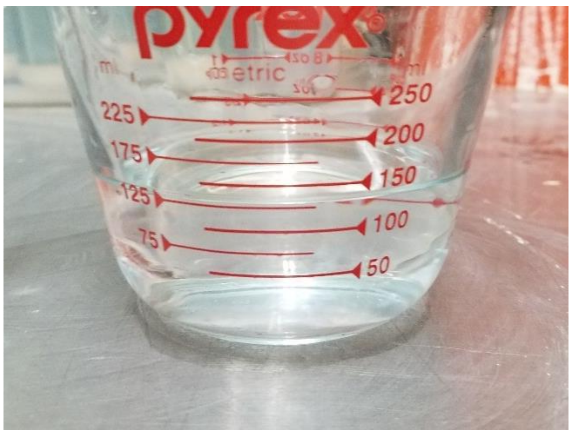

Figures 44-45. The process of the boiling water: ice water test.

Glaze Leaching Test

The fourth test, the Glaze leaching test, two cups that appeared to have issues. John Hesselberth's and Ron Roy's Ol' Blue on Porcelain at 4 percent light rutile did not pass, as well as Odyssey Clay Works' Fat Cat Red on Reclaim clay at 6 percent light rutile. Both glazes didn't appear to have any discoloration, however, the vinegar for both were noticeably cloudy. This could be from the high percentages of rutile weakening the structure of the glaze and could be leaching out a material of the glazes. Out of the sixty-nine cups left, sixty-seven passed the glaze leaching test.



Figures 46-47. Vinegar that is cloudy and contaminated.



Figures 48-49. Vinegar that is clear and uncontaminated.

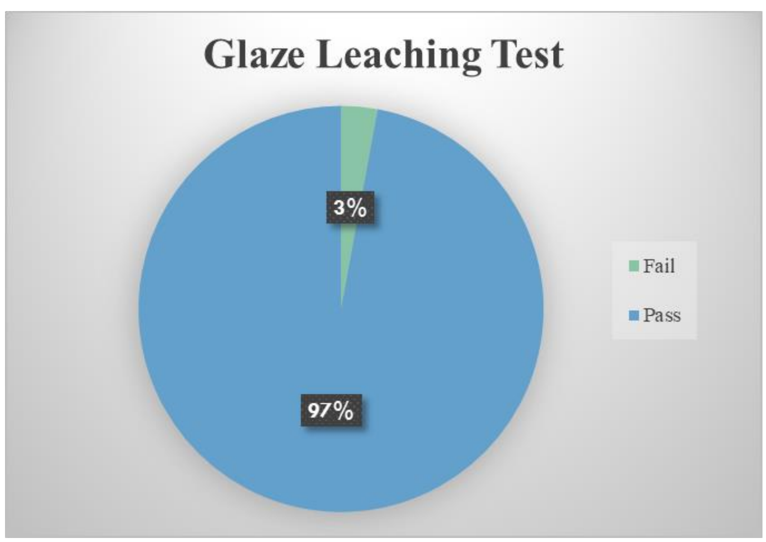


Figure 50. Graph of the pass/fail rate of the glaze leaching test

The Remaining Tests

The Lemon slice test, soda ash test, and microwave test did not disqualify any cups. There was no discoloration from neither the lemon slices nor the soda ash testing. The microwave test did not have any cups that were hotter than the water in them.

Figure 51. Cups going through the microwave test.

The Numbers

Nineteen out of twenty of the glaze tests with 0 percent rutile are food safe. John Britt's Spearmint on reclaim clay crazed. This is likely to be from a glaze and clay body compatibility rather than rutile or the glaze as a whole.

Figure 52. Food safe glazes with no rutile added.

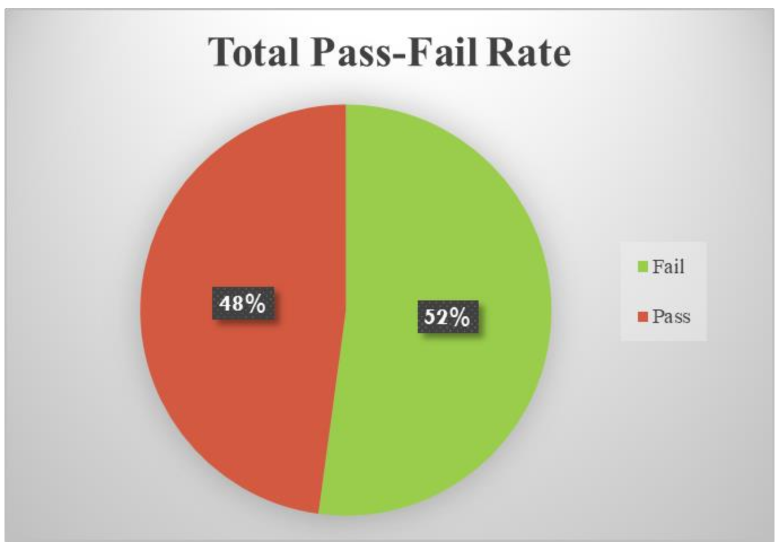


Figure 53. Graph of the pass/fail rate of total tested glaze variations.

Of the four glazes tested, John Britt's Licorice fared the worst. Of the twenty-eight variations of the glaze, eight are food safe. The base tests with 0 percent rutile and 1-3 percent light rutile on reclaim clay are food safe, resulting in only three variations that are food safe with rutile.

Figures 54-55. Food- safe cups with rutile of John Britt's Licorice glaze.

Conversely, John Britt's Spearmint has the most variations that are food safe. Of the twenty-eight cups, nineteen variations are food safe. All of the reclaimed clay and 5-6 percent of rutile on white clay did not pass the tests.

Figures 56-57. Food- safe cups with rutile of John Britt's Spearmint glaze.

The Licorice glaze had the most surprising results. Unlike the prior predictions, Licorice did not have many food-safe variations. Another surprise was Ron Roy's and John Hesselberth's Ol' Blue had no variegation, despite the additions of rutile; the glaze opacified as the rutile amount increased.

Figures 58-59. Food- safe cups with rutile of Ron Roy’s and John Hesselberth’s Ol’ Blue glaze.

Figures 60-61. Food- safe cups with rutile of Odyssey Clay Works’ Fat Cat Red glaze.

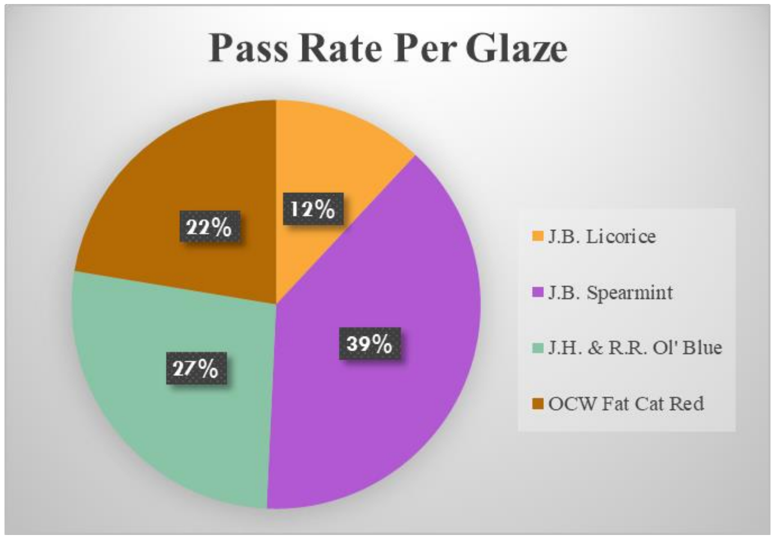


Figure 62. Graph of the pass rate of total variations by glaze

Three patterns emerged as the tests progressed. The more rutile that was added to the glazes, the more the glazes had variegation; however, at a point, the glazes lost their variegation and opacified. This point was around 4-5 percent for the glazes. These glazes opacified but were not porous, therefore they passed the tests and are food-safe.

Figure 63. Opacified but food safe glazes.

Another pattern was for the Spearmint glaze; the glaze ran down the piece as more rutile was added to the glaze. At 5-6 percent, the Spearmint glaze was running down the piece, or even off of it, rendering the cup unusable without removing the drips with diamond grit tools. This can be from the rutile adding fluxing agents to the recipe and causing the glaze to run.

Figures 64-65. Dripping glaze from 6 percent rutile.

Twenty-three of the sixty-seven food-safe cups did not have any variegation. The glaze variations are primarily from Ron Roy and John Hesselberth’s Ol’ Blue and John Britt’s Spearmint, with a single cup from John Britt’s Licorice.

Figure 66. Food safe cups with no variegation.

Conversely, of the food safe variations, sixteen variations were both food safe and variegated: nine were of John Britt's Spearmint, five of Odyssey Clay Works' Fat Cat Red, and two from John Britt's Licorice.

Figure 67. Food-safe glazes with variegation.

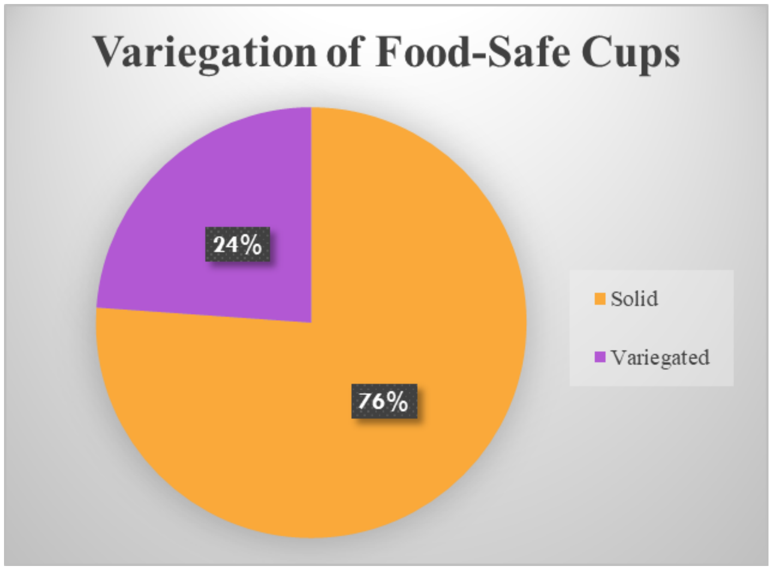


Figure 68. Graph of the percentage of variegated food-safe cups versus the solid food-safe cups.

Of the sixteen food-safe and variegated glaze variations, there were six that stood out. John Britt's Licorice on reclaim clay with 3 percent rutile had both blue speckling and brown translucency at the thin breaks in the glaze. John Britt's Spearmint on the brown clay with 4 and 5 percent rutile and 5 percent on the speckled clay were additionally strong. The 5 percent additions on both the brown and speckle clays appear with light green speckling concentrated on the bottom portion of the glazes as gravity is pulling the glaze down as it is molten at peak temperature. The 4 percent rutile variation has less of the light green speckling and appears light green at thin areas of the glaze.

Continuing, Odyssey Clay Works' Fat Cat Red on brown and porcelain clay with 1 percent rutile added are good options as well. On the brown clay, the glaze appears warmer from the color of the clay and there is light purple speckling in thicker areas of the glaze. On the porcelain clay, the glaze appears dark maroon with light purple speckling. The glaze appears light orange where the glaze is thinner.

Figure 69. Best variations of variegated, food-safe glazes.

Summary

This experiment explored the addition of rutile in food-safe glazes. Four glazes were tested on five common clay bodies, testing seven percentages of rutile, totaling 140 variations. After the visual inspection, one hundred made it to the cutlery marking test. Thirty-one cups did not pass the cutlery marking test, leaving sixty-nine variations for the next tests. Only two cups were eliminated from the glaze leaching test- sixty-seven cups passed all remaining tests and were deemed food safe. Of the food-safe glaze variations, Ron Roy & John Hesselberth's Ol' Blue had no variegation with the addition of rutile. Additionally, light rutile appears to be incompatible with John Britt's Licorice glaze as all but eight variations failed the tests.

The various possibilities of rutile in food safe glazes were only limitedly studied in this experiment. Variegation comes with a higher probability of issues and defects within the glazes, lending itself to constant testing to determine the food-safe quality of the glaze. Food-safe glazes prevent bacteria, mold, and toxic materials from accumulating on or contaminating the glazes, which can subsequently be consumed. Ceramic glazes are essential in sealing the surface of the clay and are often used to decorate ceramic works. It is important for a potter to find the balance between decoration and food-safe qualities when choosing the glaze for work that is designed to be used for food.

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Appendices

All recipes show ingredients by percentage

Appendix 1

<u>John Britt's Licorice</u>

Custer Feldspar	20
EP Kaolin	20
Frit 3134	20
Silica	19
Wollastonite	15
Talc	6
Red Iron Oxide	9
Bentonite	2
Cobalt Carbonate	1

Appendix 2

<u>Odyssey Clay Work's Fat Cat Red</u>

Custer Feldspar	31
Whiting	21
Silica	18
EP Kaolin	9
Frit 3134	9
Gerstley Borate	8
Talc	4
Tin Oxide	5
Chrome Oxide	.2

Appendix 3

Ron Roy's and John Hesselberth Ol' Blue

EP Kaolin	30
Wollastonite	29
Frit 3195	20
Silica	17
Nepheline Syenite	4
Copper Carbonate	3
Cobalt Carbonate	1.5

Appendix 4

John Britt's Spearmint

Kaolin	28
Wollastonite	28
Frit 3134	23
Silica	17
Nepheline Syenite	4
Copper Carbonate	4
Bentonite	2

Appendix 5

Porcelain Body

Custer Feldspar	25
EP Kaolin	25
OM4	25
Silica	15
Nepheline Syenite	10

Appendix 6

White Body

OM 4	25
Custer Feldspar	25
EP Kaolin	20
Silica	17.5
Nepheline Syenite	12.5

Appendix 7

Speckle Body

OM 4	25
Custer Feldspar	25
EP Kaolin	20
Silica	17.5
Nepheline Syenite	12.5
Granular Ilmenite	2
Red Iron Oxide	.5

Appendix 8

Red Iron Oxide (Brown) Body

OM 4	25
Custer Feldspar	25
EP Kaolin	20
Silica	17.5
Nepheline Syenite	12.5
Red Iron Oxide	3