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The Dreaded Orange Spot

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Where do they come from and, even more importantly, is there anything that can be done to prevent them?

The soap was perfect. It had traced and poured without incident, it had unmolded easily, and its creamy color and texture spoke to my unrivaled saponifactory skill. After curing for four weeks it was gentle on the skin and pleasing to the nostrils. All was right with the world.

Vial with soap sample in place

Then they showed up. Small at first, really unnoticeable to anyone but an anal-retentive lavanteur, they took up residence on a few bars that would now be destined for my own personal use. Not that there was anything wrong with the soap, goodness no. The blemished bars would perform every bit as well as their fair-complexioned siblings. But when I send my soaps out into the world I want them to make the best possible first impression and so it is best to keep the spotty ones at home in the cupboard. No harm done.

But as these bars lost the blush of youth and entered their adolescence, most of them developed orange zits of one kind or another. Unlike human teen-agers, however, they would never outgrow them. I have come to understand through reading, both in the industrial and the craft literature, that the Dreaded Orange Spots (DOS) are caused by the oxidation of "soft" oils like olive oil. Industrial soapmakers have largely given up on soft oils in favor of coconut oil and tallow. But the diversity of modern handcrafted soaps owes much to the availability of a dozen or more soft oils that are routinely used to lend their specific properties to soap. In the fall of 2004 I began investigating the causes and cures for DOS along with my chemistry students at Hampden-Sydney College.

The Experiment Equipment

Industrial soapmakers use a variety of techniques for measuring the rancidity of oils, chiefly the rancimat method. But I wanted a method that most handcrafted soapmakers could use without expensive or specialized equipment. We used an ordinary flat-bed scanner and a free computer program called the Gimp (available online at www.gimp.org).

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This study on the cause and prevention of the Dreaded Orange Spot was conducted by Stephen Diegelman, Shea Duerring, Michael Kraemer, Jamie Rock, and Keith Williams under the direction of Kevin M. Dunn at Hampden-Sydney College.

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DOS *- Con't from front cover*

The scanner would be used to acquire digital images of our soaps over several weeks and the Gimp would measure their color.

To speed up the arrival of the spots, we stored our soaps in an incubator at 60°C (140°F). For each of our soaps, we monitored the color of two samples, one stored in a vial of oxygen and the other in a vial of nitrogen. Comparing these two samples would reveal the role of oxygen in the development of soap rancidity. A specialized holder was developed which left the soap in contact with the gas in each vial while allowing the soap to be scanned without removing it from the vial.

Soap holder made from a disposable dropper

A disposable medicine dropper available from Cynmar (www.cynmar.com) was formed into a sample holder. The bulb of the dropper has been snipped off and the remaining cavity filled with a one-gram sample of soap. A plastic collar keeps the sample holder from sticking to the sides of the vial.

The sample holder is placed in a small vial filled with either oxygen or nitrogen gas. During incubation, the vials are stored cap-down, leaving the soap in contact with the gas in the vial. For scanning, however, the vial is placed right-side-up on the scanner, making it possible to scan the soap sample without removing it from the vial.

The recipe used:

100.0 grams olive oil
13.0 grams sodium hydroxide
26.0 grams distilled water
0.1 grams of any chosen additives

Sodium hydroxide was dissolved in water, allowed to cool, added to the oil, and shaken continuously for 15 minutes. It was then poured into a mold and kept in an incubator for 4 hours at 60°C (140°F). Previous tests had shown that saponification is complete at the end of this incubation. One-gram samples of each soap were then placed in vials as described above and the soap was artificially aged by incubating it for several weeks while scanning it on a daily basis to record its color. The color was measured using the Gimp "color picker" tool, set to measure "color saturation." Soap is noticeably orange when its color saturation is above about 30%.

Reproducing Orange Spots at Will

The first question to ask is "Can you produce orange spots under these conditions?"

Two curves are shown on the color-saturation graph for pure olive-oil soap. The color saturation curves show what happens to the color of the soap as it ages. The lower curve is for soap stored in nitrogen, the upper curve for soap stored in oxygen.

The lower curve shows that the soap becomes gradually more colorful (orange), rising to 38% saturation after 300 hours of incubation. The upper curve shows that the soap stored in oxygen turns orange more rapidly than that stored in nitrogen. Of particular note, there is a rapid increase in color saturation at about 75 hours.

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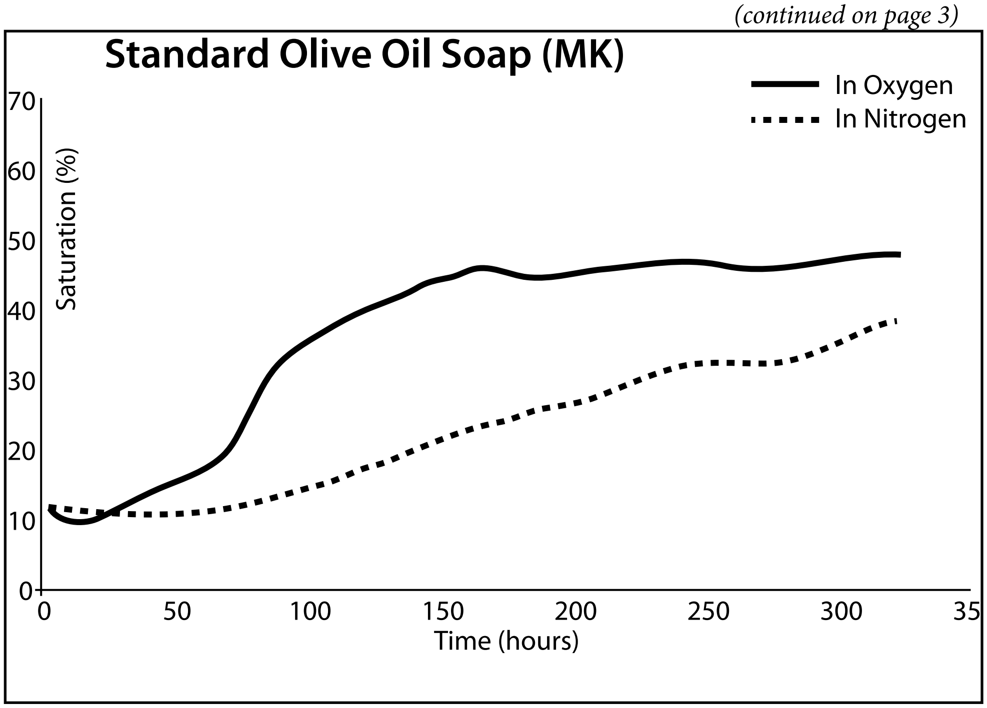
Scientific Method : Change One Thing At A Time

When cooking or soap-making it is human nature to give free reign to our creative impulses. Though we may be careful to follow a tried-and-true recipe for production-grade soap, we may develop new recipes by making several variations at the same time. To answer questions scientifically, however, it is best to change only one thing at a time.

In the present investigation, all soaps are subtle variations of one soap recipe.

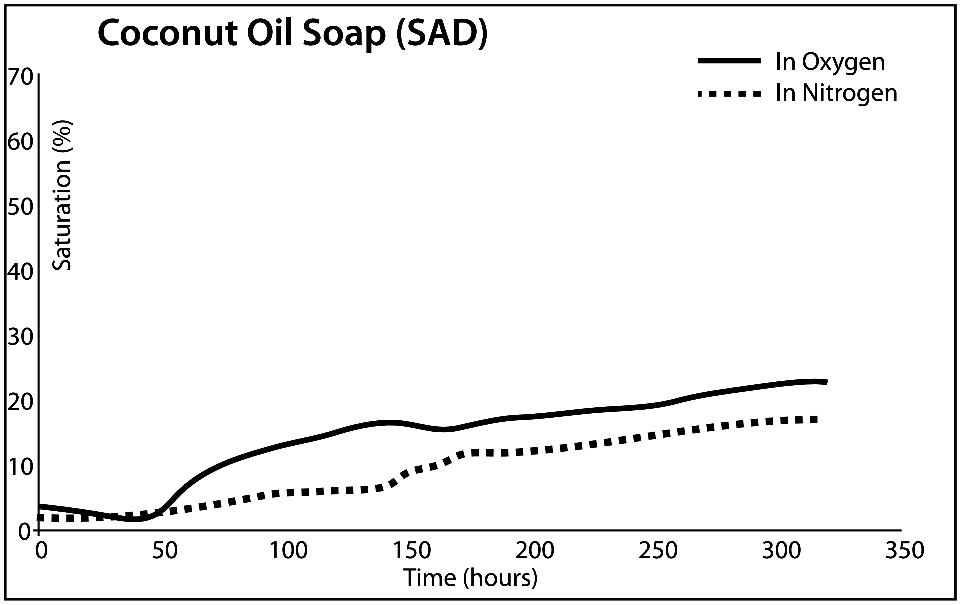


The "Induction Period"

The time at which such a rapid increase occurs is referred to as the "induction period." A soap that turns orange rapidly will have a short induction period; a soap that stays white for a long time will have a long induction period.

The color saturation curve for coconut oil shows that it is relatively resistant to rancidity. For one thing, there is little difference between the color of soaps stored in oxygen and nitrogen. Neither soap rises above 20% saturation.

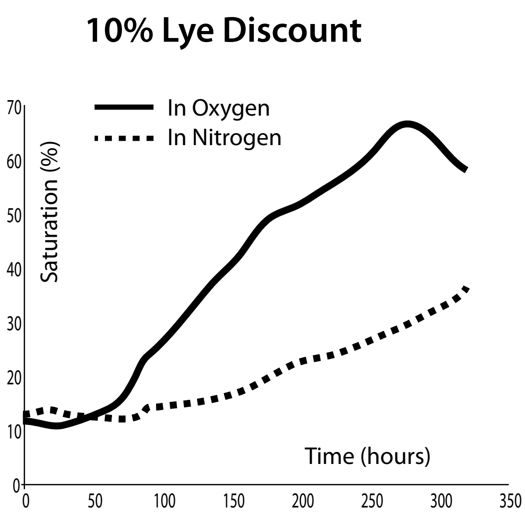
If all of our soaps behaved as coconut-oil soap does, we would never see orange spots.

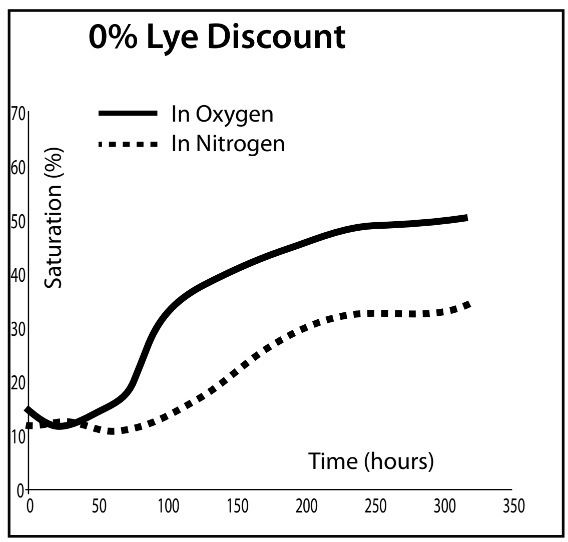


Now that we know how to measure orange spots, we can try to prevent them by changing one thing at a time in the soapmaking process and seeing how these changes effect the induction period and the final color saturation.

Is It The Lye Discount (Superfatting)?

It is sometimes asserted that an excessive lye discount causes orange spots. We therefore made two soaps identical to our standard soap except that in one case the lye discount was 10%, in the other, 0%.





From the color saturation curves in the two following graphs, we can see that, indeed, the 10% discount soap does eventually become more orange than the 0% discount soap. But looking more closely we also see that the induction period is about the same, 75 hours.

Even at 0% discount the soap becomes noticeably orange in about the same time as at 10% discount. Since our goal is to prevent orange spots altogether, we should not look to changing lye discount as a cure for them.

What about additives?

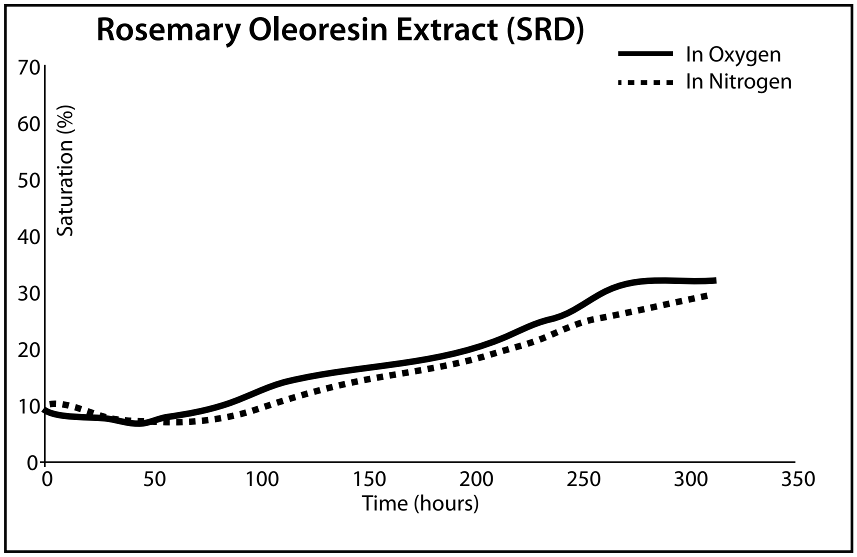
Some handcrafted soap literature has suggested several different additives as preventatives for orange spots. We tested the most commonly suggested additives using 0.1 grams of each additive for 100.0 gram soap, i.e., the additive was 0.1% of the oil weight.

The test results showed that the color saturation curves for **Grapefruit Seed Extract**, **Vitamin C**, **Vitamin E**, and **Sodium Citrate** are virtually identical to that for pure olive oil soap. They showed no prophylactic effect in our tests.

Butylated Hydroxytoluene (BHT) lengthened the induction period to 150 hours, twice the induction period of pure olive-oil soap; it would appear to be an effective preservative for handcrafted soap.

Two other additives, **Rosemary Oleoresin Extract (ROE)** and **Sodium Ethylenediammine Tetraacetate (EDTA)** showed almost identical performance. Both lengthened the induction period beyond 300 hours (the limits of our test) and held the eventual color saturation below 30%.

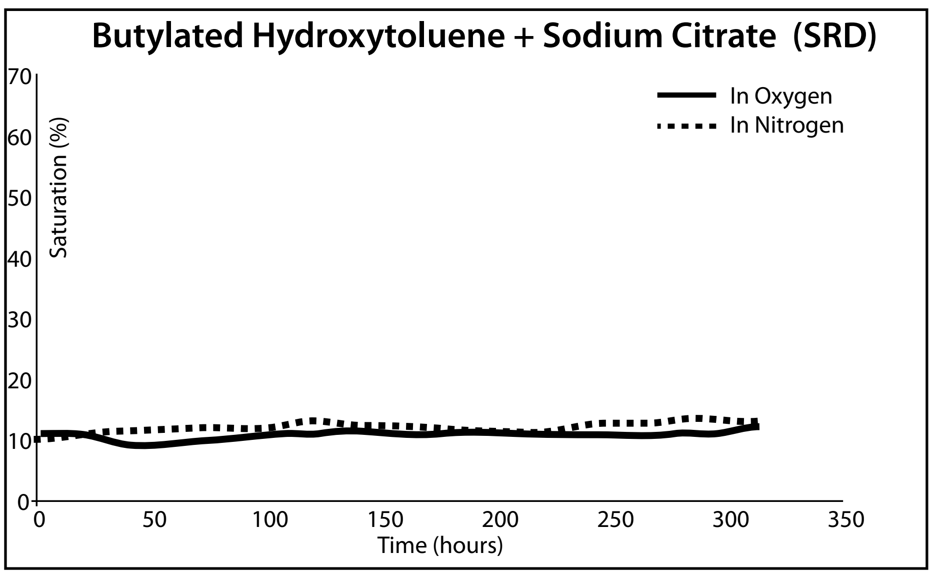
Thus ROE and EDTA showed the best preservative effects of any single additives in our tests (see graph to left).



Do additves do better when combined?

We next looked at combinations of additives. Vitamin E (0.1 g) + Sodium Citrate (0.1g) performed slightly better than Vitamin E alone, that is to say, it did not perform very well. ROE + Sodium Citrate actually performed worse than ROE alone. Three combinations, however, performed dramatically better than any single additive. BHT + EDTA and ROE + EDTA each lengthened the induction period beyond 300 hours and held the eventual color saturation below 25%.

The best combination in our tests, however, came from BHT + Sodium Citrate. Used together they lengthened the induction period beyond 300 hours and held the eventual color saturation to about 10%, the same color as fresh soap.



As a result of our tests we suggest that by using 0.1% of your oil weight of BHT and Sodium Citrate you may altogether prevent the appearance of DOS. Inevitably, however, we will run into the ongoing, quasi-religious debate between the use of natural and artificial additives. A full discussion of that debate is probably better left for another article.

Suffice it to say that while I, personally, have no qualms about the use of artificial additives, there are many in the handcrafted soap community who do. In particular, many of your customers may value natural ingredients, perhaps even insisting on them. I believe that one of the chief advantages which the handcrafted soapmaker has over the industrial soapmaker is the ability to tailor soap recipes to the preferences of individual customers.

If your client base is heavily tilted toward natural ingredients, using 0.1% of your oil weight in ROE is very likely to significantly extend your shelf life. If, on the other hand, your client base is indifferent to the natural/artificial distinction, the combination of BHT and Sodium Citrate is likely to out-perform ROE as a preservative.

Whatever you decide to do, I would urge you to accurately label your soaps. People have myriad reasons for their preferences, some due to medical conditions, some due to allergies, some due to crackpot theories or personal whims. They have a right to those preferences, whatever their reasons. Your only choice is whether or not to serve customers with those particular preferences.