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The sweating of lipsticks

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Theory of sweating

Sweating, or the excretion of oil on the surface of a lipstick/hot pour product, is a problem that has confronted this company for many years. Every lipstick that was sweating was examined and analysed using infrared spectroscopy. In all cases the oil excretion was found to be pure ricoil (castor oil), lacking any trace elements of other components.

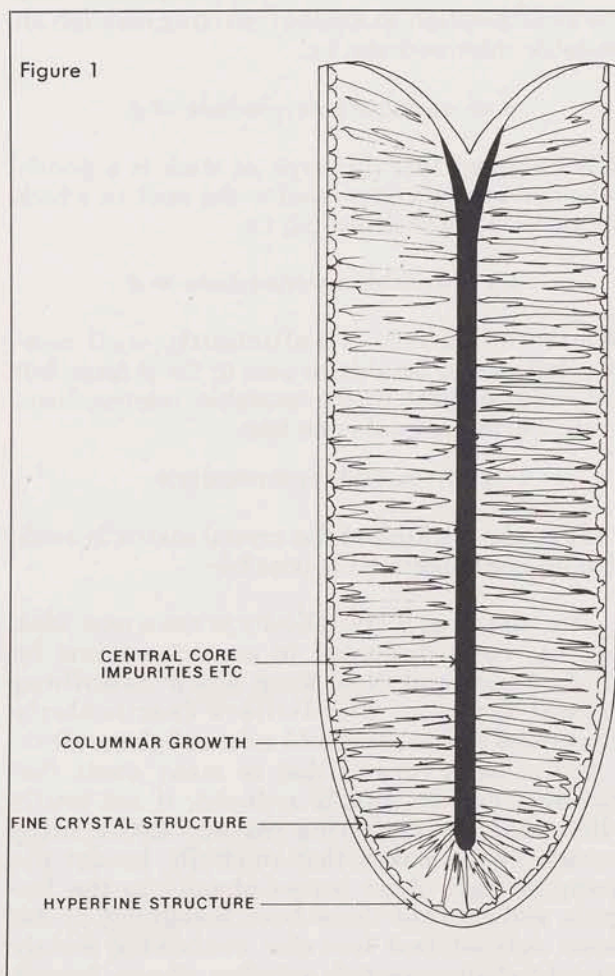
There is one cause and one cause only for this sweating, and that is an imbalanced formula. All theory relating to sweating must therefore be understood to relate to a poor formula. Some waxes tolerate more ricoil than other wax bases before sweating occurs, and the better the formula the higher the percentage of oil that can be included.

It has become increasingly obvious, the more that lipstick formulae have been examined, that the crystal lattice formed by the major waxes once the moulding has set is not static, nor is it homogeneous throughout the stick. It is also believed that the outer skin of the lipstick is under considerable stress as a result of the sudden cooling experienced in the initial contact of the mass with the mould, and that this stress situation is further increased by the process of flaming, in which the top surface of the stick is rapidly melted and cooled to give a hyperfine crystal structure. The crystal structure on the surface is so fine that the boundary between crystallinity and amorphousness is no longer clear. It is the hyperfine structure that gives the appearance of gloss.

The structure envisaged is similar to that of a metal casting that has been tempered. The tempering is equivalent to the flaming, which gives the hypofine structure that increases in crystal

size to a fine structure through to a medium structure and from there through a columnar growth type of structure to the centre where the

Figure 1

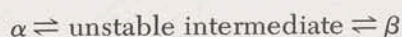


impurities, if any, will exist. (See figure 1.) This situation has been verified by the outer skin being harder than the centre, which tends to be slightly mushy.

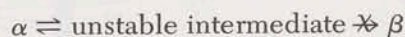
Sweating is a seasonal phenomena (under ambient conditions) and is usually observed after a large temperature fluctuation, or increase in temperature, taking place over a short period of time. This is normally associated with a "muggy", high relative humidity day, although studies of barometric pressure and relative humidities over a four month period in 1975 failed to produce any significance between sweating and humidity. It is believed that in the cool winter and spring months the crystal structure within the stick is fairly static and the wax and ricoil are in stable equilibrium.

However, with the coming of summer the temperature fluctuations become greater. With increasing temperature the wax lattice modifies (in an imbalanced formula) and the wax/ricoil equilibrium is altered so that the ricoil is not held by the lattice, and the ricoil begins to migrate to the surface of the stick. It is possible that it takes a number of cycles before the ricoil becomes fully disbound from the wax matrix, since a reequilibration will take place in the evening as the temperature cools.

It may also be observed that the ricoil sweats and returns to the stick, despite the temperature not having fallen, and this is believed to occur because the wax matrix or lattice passes from one configuration to another, passing through an unstable intermediate; i.e.



It is believed that this type of stick is a poorly balanced formula compared to the stick in which the ricoil is not reabsorbed; i.e.



where the matrix is sufficiently well constructed, not to be able to pass to the β form, but can only readjust to the unstable intermediate. In the perfect formula one has:



That is, the packing of the crystal matrix is such, that no rearrangement is possible.

The idea of α , β crystallinity is not a new idea, and has been discussed in emulsion terms by E. A. Flack and Nils Krog when describing crystal forms of emulsifiers (particularly glycerides and various acid esters of glycerides).

It has been proved that in many cases that sweating can be greatly reduced, if not totally eliminated, by modifying the wax lattice using branch chain waxes that sterically hinder the many straight chain waxes already in the formula and prevent them from realigning. It has been noticed that formulae containing mainly straight chain materials and few, if any, branch

chain materials have two tendencies. First, they sweat profusely after a few minutes at 40°C and this sweating is reabsorbed after a short time. Second, the surface gloss on flaming is poor, and this partial gloss diminishes with passing time. This property is probably due to wax matrix modification at the surface. Waxes such as microcrystalline waxes and polyethylene wax have been found to be notorious for wax lattice modification after moulding.

The use of oxidised microcrystalline and oxidised polyethylene waxes greatly improves the wax lattice stability, which is reflected in the reduction of sweating and also in the better and longer lasting gloss obtained. It is also noticed that the reabsorption of sweating at 40°C is considerably reduced, and that reabsorption takes place on cooling the stick back down to ambient temperature.

The inclusion of heavy branch chain waxes (C_{18} - C_{32}) such as triglycerides of fatty acids and ethylene glycol esters of fatty acids in combination with branch chain oils has greatly reduced and in many cases eliminated the incidence of sweating. The wax matrix has been so modified that no realignment or transmutation is possible, a theory that is supported by the high gloss and the long-term stability of this gloss at 40°C storage.

Support for the lattice modification theory can be found in the examination of penetration data. In some cases the original synthetic straight chain formulae underwent such drastic lattice modifications that they became so brittle that they cracked and fell apart after a few hours, despite the fact that they were ideal texturally when removed from the mould. Penetration readings altered by a factor of ten overnight.

In the ideal formula, where the wax matrix has had the correct steric hindrance, and where the molecular packing is correct, the hardness after three hours of moulding is the same as the hardness six weeks later. However, it is not as simple as it might appear to formulate the ideal lipstick formula. Certain materials by nature of their chain length or chemical nature have adverse effects on the formula, which either require careful balancing or in some cases absolute exclusion. Nor is it easily proved scientifically which materials have an exact effect, since the removal of one material means that it must either be replaced by another, or the formula must be corrected back up to 100% to compensate for its removal.

Therefore, in the light of the preceding paragraph, (although I am quite willing to be proved incorrect), the following materials were found to be impossible to incorporate into the type of system being examined, without causing a dull flamed surface: wax OP, wax OM, wax VPW SW, wax RT, wax BJ, wax FL, (Montan wax derivatives) and cetyl alcohol. These materials must be considered as lattice inhibitors. Stearic acid has been used in very glossy stick finish formulae, but it has caused severe sweating that could not be eliminated by formulation.

Influence of colour concentrates

The colour concentrate consists of pigment or dyestuff lakes dispersed in ricoil. Every pigment and lake has its own associated oil absorbency. In most formulae the colour concentrate level plus the ricoil level accounts for about 50% of the formula (in the ideal formula it is exactly 50%). It has been noticed that where a combination of colour concentrates exceeds 20%, the likelihood of sweating is greatly increased, and the formula is virtually impossible to compensate. (The effect of high levels of individual concentrates has not been examined.)

In the ideal formula the risk of sweating is slight, and that risk is further reduced by adding a "hardening" base of branched chain waxes. Nevertheless the risk still exists at 40°C testing. It has also been observed that high concentrate levels can be responsible for a variation in texture, that is, lack of rigidity, softer, or mushier, when compared to a stick of lower concentrate level. It is concluded that the oil-binding affinities of the different pigments and lakes interfere with the lattice formation of the wax base. Thus when the ricoil, though weakly bound to the pigment, is added to wax base as a concentrate, it does not become associated with the wax base, but remains weakly bound to the pigment in an interstitial placing in the matrix. Thus the ricoil is less tightly bound than had it been added directly to the wax base without the pigment.

The interstitial placing within the matrix is tolerable at lower (up to 20%) levels, but at higher levels produces incoherence within the binding forces of the lattice, resulting in a softening of the stick. Naturally, with the binding forces reduced and the basic lattice distorted by pigment, the ordered balance of the wax base is partially destroyed and realignment or transmutation of the lattice can take place, so that sweating may once again be possible. It is highly likely that it is the ricoil from the concentrate, which we believe would be less highly bound than the ricoil in the wax base, that migrates. Without the use of sophisticated labelling techniques there is no way of telling.

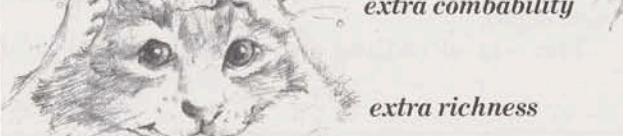
Addition of more of the lattice-forming waxes, together with branched chain waxes, does partially restore the structure. It is also of interest to note that the higher concentrate level sticks are marginally less glossy after flaming than the lower concentrate level ones.

Influence of dry pearls

The inclusion of titanium coated mica type pearls has been found to increase the tolerance of the wax base to higher concentrate levels. There is no interaction of the pearl with the wax lattice, and it is thought that it takes up a position within the interstitials of the matrix and acts as a "sponge" for the unbound ricoil, which might otherwise have migrated to the surface as sweating had the pearl not been present. This "sponge" effect, however, is not extremely

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efficient and should not be used as a means of incorporating extra concentrate. Addition of large quantities of pearl does tend to give an unpredictable drying effect to the stick's textural application. The exact function of the pearl in the lattice is not known, nor is there a great deal of data assembled at the present time. It is hoped that future research will yield greater information.

Effect of moulding

Mould lubrication, when lightly applied, plays little part in the effect of sweating. However, with increasing lubrication of the mould there is an increased tendency for the lipstick to sweat at 40°C testing. It has been observed with excessive oiling that a previously good batch of stable formula sweated after 24 hours at an increasing ambient temperature, whereas the same batch from a normally oiled mould was stable at 40°C testing.

This effect is attributable to the lipstick mass being a homogeneous mixture of waxes and oils. When this mixture hits the side of the mould it solidifies, but also takes into solution (partially at least) some of the excess lubricant oil present. Thus the oil to wax ratio at the surface of the stick has been increased. This may be in excess of the stable equilibrium, and sweating may well occur.

The rate of cooling also has an effect, but at

the laboratory rate or production rate it plays no major role. Only slow controlled cooling at an unrealistic rate produces a grainy stick that is more likely to sweat.

Effect of delay times

Delay times are the periods of waiting between moulding and flaming and between flaming and testing. All the most stringent tests were done with virtually no delay time, which was found to be very severe when testing for sweating. Thus a stick was moulded, flamed, and tested at 40°C without a stop.

In a well-balanced formula the delay time has little or no importance, but the marginal formula benefits from a delay time of a few hours or more, when it has been observed that sweating is reduced if there is a delay before flaming. Whether this is due to an equilibration of the lattice coupled with an increased wax to oil stability can only be surmised.

It has also been found in borderline formulae that the type of sweating observed varies, depending on the delay time between flaming and testing at 40°C. Immediate testing of the flamed lipstick at 40°C produces a fine, misty sweating. However, if a delay of a few hours is made prior to testing, then the sweating appears to be coarser, in the form of minute droplets evenly distributed across the stick. Furthermore, if this stick is left flamed for 24 hours before testing, then the sweating appears as coarse, large, irregularly spaced droplets, normally at faults or at the seam of the stick.

This delay after flaming obviously allows some rearrangement of the surface, which is indicated by the larger droplets of sweating occurring. It would seem that the hyperfine structure at the surface modifies with the passage of time to a fine structure. Thus instead of a very high stress over the whole surface, this breaks down to a lower stress with a larger crystalline surface structure.

It has been noticed that if a newly flamed lipstick is "scuffed" at a small area then sweating occurs exclusively at the surface disturbance. This factor is good evidence that surface stress is especially present after moulding and flaming. In general, sticks that were unflamed showed less tendency to sweat, but observation was difficult.

Conclusion

Much of this paper is based on experience and supposition. The supposition predicates a physical picture that has been built and destroyed many times, each new picture being an advancement from the last. The final conclusion is a picture that fits the cases examined throughout all our brand and experimental sticks: a picture of a "breathing" lattice kept in one form by means of steric hindrance and careful wax balancing—that is, molecular packing. There is still much to be learned, and it is hoped that this paper will help future development.

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