Process and Material Considerations in the Industrial Application of Lubricants in Rigid PVC Extrusion

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The proper lubrication of rigid PVC compounds often plays an important role in the successful operation of a rigid PVC extrusion plant. Changes in a particular lubricant system can have a substantial effect on the fusion rates of a rigid PVC compound and on the thermodynamics of the fusion process. Recently published research on the morphology of PVC resin and rigid PVC finished products has provided new insights into the morphological changes rigid PVC undergoes during processing. Lubricants can have a substantial impact on the rates and extent of morphological changes of rigid PVC during processing. Successful industrial optimization of a lubricant system involves balancing the rheological characteristics of the compound against the shearing and heating characteristics of the extruder and die tooling. Changes in the extruder and die tooling or lubricant system will result in changes in the work and energy balance. The impact of increases in fusion promoting lubricants, as well as increases in fusion delaying lubricants, on the work and energy balance for a twin screw extruder is explained.

INTRODUCTION

The proper lubrication of rigid PVC compounds often plays an important role in the successful operation of a rigid PVC extrusion plant. A rigid PVC processor first selects a PVC resin, stabilization system, and other necessary compound ingredients which, when properly compounded and processed, will meet the performance and economic demands of the finished product. He then will search for a suitable lubrication system which will allow the selected compound to be efficiently processed on the equipment available. As shown in *Fig. 1*, lubricants can be considered an important side of a triangle of fundamental processing parameters for rigid PVC products. A great deal of compound development time is often spent trying to find a suitable lubrication system for the PVC compound and equipment of interest. Lubricants are key ingredients in any rigid PVC formulation. In general, a well balanced lubricant system becomes more important as productivity demands increase and as efforts are made to decrease the raw material cost of the compound.

Over the past few years, great advancements have been made in understanding PVC resin morphology and the morphology of various PVC end products. This research is providing new insights into the rheology and lubrication of rigid PVC compounds. Recently published works by numerous authors clearly indicate that the morphologies of rigid PVC end products are quite complex and dependent upon processing history (1, 2, 4, 6, 9, 10, 16). Some products which are processed at relatively high temperatures, such as rigid, clear PVC sheet, film and bottles, are very well fused. Other products, perhaps the majority of extruded and injection molded opaque rigid PVC products, are very often processed at lower temperatures. These products, which include PVC pipe, vinyl siding, and PVC window profiles are not always as well fused and retain more of the particulate characteristics of PVC resin. Some recently published works indicate that for some compounds the optimum balance of physical properties is achieved with an intermediate degree of fusion (6, 10). The performance requirements for many products can be met with various degrees of fusion, however, it is reasonable to expect that optimum product quality is achieved at an optimum degree of fusion.

In the rigid PVC industry, there is a wide variety of PVC resins and compounds, and a wide variety of processing equipment. Rigid PVC can be a difficult and sensitive material to process. In some formulations, a slight change in the lubricant balance can have a dramatic effect on the productivity of an operation and the physical properties of the end product. This paper will discuss, using a simple lubricant system as an example, some important process and material considerations when evaluating lubricants in a torque rheometer and during their application in twin screw extrusion.

CHARACTERISTICS OF PVC RESIN

The particulate nature of PVC resin is shown in *Fig. 2*. PVC consists of resin grains which average approximately 130 microns in diameter; these are composed of subgrains of 40 microns diameter; subgrains consist of agglomerates of primary particles of 0.7 micron average approximate size. The primary particles appear to be the primary flow units during melt formation and fusion of rigid PVC. Primary particles consist of domains of 0.2 micron average

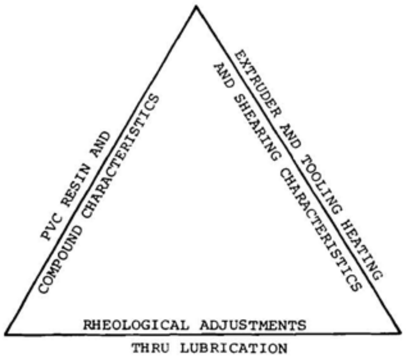

Fig. 1. Fundamental rigid PVC processing parameters.

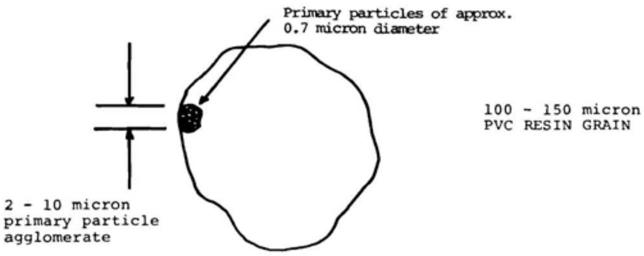

Fig. 2. Structure of PVC resin.

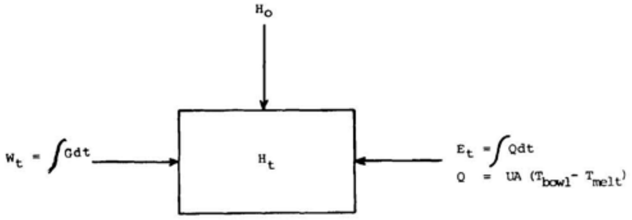

Fig. 3. Simple energy balance for a torque rheometer.

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* *Nomenclature for Fig. 3*

$$H_t = H_o + E_t + W_t$$

$$T_{melt,t} = H_t/m\,C_p$$

$$H_o = m\,C_p\,T_o + P_oV$$

Where:
H_o = Initial enthalpy of material, BTU
H_t = Enthalpy of material at time t, BTU
E_t = Amount of heat transferred at time t, BTU
W_t = Amount of work generated at time t, BTU
m = Charge size, lb.
C_p = Heat capacity of material, BTU/LB°F.
P_o = Pressure upon charging, psi
V = Bowl volume, FT^3
T = Temperature, °F.
t = Time, HRS.
Q = Rate of heat transfer, BTU/HR
U = Overall heat transfer coefficient, BTU/FT^2 °F.HR
A = Bowl surface area, FT^2
T = Temperature, °F.
G = Torque, FT LB.

size, which, in turn consist of microdomains of 0.02 micron size. The microdomains are the smallest particles so far identified. The domain structure is thought not to be deformable during processing (16).

TORQUE RHEOMETER EVALUATIONS OF LUBRICANTS

The torque rheometer is a useful and widely accepted tool for screening rigid PVC formulations. *Fig. 3* is a simple energy balance for a torque rheometer. Basically, the test procedure involves compaction of the test material to a fixed volume, followed by mixing and heating. The amount of work going into the material is indicated by the torque necessary to turn the mixing blades. The amount of heat transferred to the material is mainly a function of material and rheometer bowl temperatures.

Figure 4 represents a typical low temperature torque rheometer curve for a PVC pipe formulation where the level of paraffinic lubricant is varied within the working range of the formulation. Initially, the material is fully compacted in the torque rheometer bowl, causing high initial torques which rapidly decrease as resin grains are compacted into smaller particles. The torque generated during this compaction stage is reduced with increasing levels of paraffinic lubricant. This compaction torque level continues as frictional heat is generated and heat is transferred from the bowl until the temperature of the material reaches a point where the particles begin to stick together initiating an increase in torque. The rate at which the torque increases is also reduced with increasing levels of paraffin lubricant.

Wingrave's work suggests that each torque transition in the torque rheometer can be related to changes in the particulate nature of the melt (1, 2). For a simple PVC pipe compound, he developed a mathematical model of the torque rheometer fusion process based on random particle size changes from larger to smaller particles. His model, as well as work by Allsopp (4), suggests a PVC fusion mechanism for a torque rheometer as shown in *Fig. 6* where PVC resin grains undergo a compaction and densification process yielding individual primary particles and primary particle agglomerates which eventually fuse together. As mixing continues, the particulate nature of the melt is gradually eliminated, provided a sufficient temperature is attained.

Calcium stearate, as shown in *Fig. 5,* has an opposite effect on initial torques than that of paraffinic lubricant. As its usage level is increased, the amount of torque generated in the initial stages of fusion increases. Thus, the amount of time necessary for the temperature of the melt to reach the point where the next series of particulate changes are initiated is decreased. Also, once a fusion transition initiates, increases in the amount of calcium stearate increases the rate of propagation of that transition.

Referring back to *Fig. 3,* increases in the level of paraffinic lubricant, a fusion delayer, results in the energy required for fusion to be gained more through heat transfer and less through work input. Increases in calcium stearate level, a fusion promoter, increases the amount of work imparted to the material and decreases the amount of heat transfer necessary in order to attain the temperature at which fusion is initiated.

LUBRICATION OF TWIN SCREW EXTRUSION FORMULATIONS

In most PVC pipe operations, the effects of the lubricant system on the fusion characteristics of the dry blend are of much greater importance than the internal or external functionality of the lubricants. Obtaining an adequate amount of fusion in the end product is very important to insure adequate hydrostatic and impact performance. For a given extruder, the optimum lubricant balance occurs when the rheological characteristics of the compound, as determined by the lubricant system, accommodate the compression and shear characteristics of the extruder screws and die tooling. A well balanced lubricant system results in desirable extruder loads, high output rates, good dimensional control, and good finished product physical properties. An imbalanced lubricant system can result in low output rates, high or low extruder loads and barrel temparatures, rough surfaces, low impact strength, midwall tears or midwall voids, and poor dimensional control. Overall, a poor lubricant balance is a production manager's nightmare.

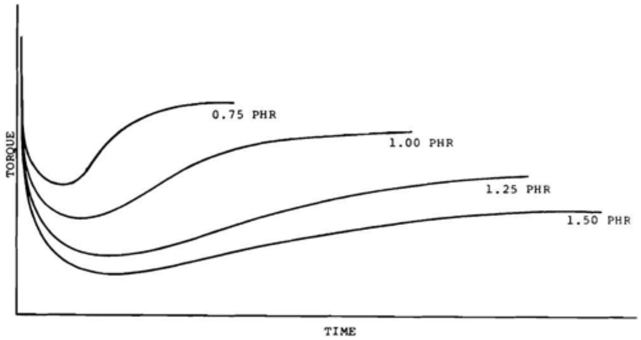


Fig. 4. Effect of paraffinic lubricant levels on low temperature torque rheometer fusion characteristics.

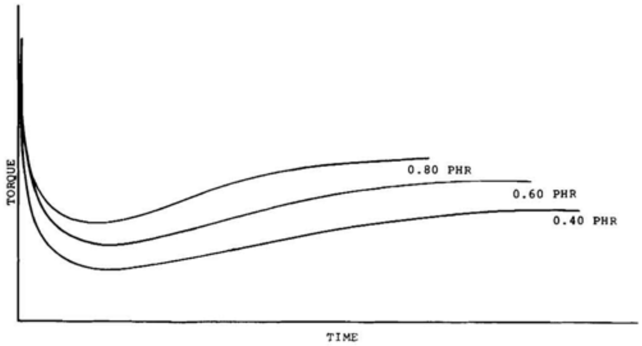


Fig. 5. Effect of calcium stearate levels on low temperature torque rheometer fusion characteristics.

Figure 7 shows a simple energy balance for a twin screw extruder. Basically, PVC compound is fed into the extruder and is gradually heated and compressed. Then the material enters a decompression zone and is devolatilized. After devolatilization, heating continues as the material enters another compression zone and finally the metering zone. By this time, the material is nearly fully compressed and heat, in many cases, is being removed through barrel and screw cooling. As the material is leaving the screw tips, it is at its highest pressure and is fully compressed. The screw shear imparted to the material at this stage is thought to have the greatest effect on the degree of fusion obtained in the finished product (13). Variations in the amount of heat transferred from the barrel surfaces and the amount of work generated can significantly affect the degree of fusion obtained in the end product. For example, Benjamin (10), in his study of effects of processing on the physical properties of PVC pipe, obtained pipe samples from the same extruder with fusion levels, as determined by a capillary rheometry test method, of 32 through 90 percent by varying the extrusion conditions. These pipe samples had considerably different physical properties; the optimum combination of impact and tensile properties occurred at a fusion level of about 60 percent.

Allsopp (4, 15) describes the mechanism of fusion in extrusion as a "CDFE Mechanism", i.e. compaction, densification, fusion, and elongation. This mechanism is schematically shown in *Fig. 8.* Essentially, as the material is heated and compressed, this mechanism

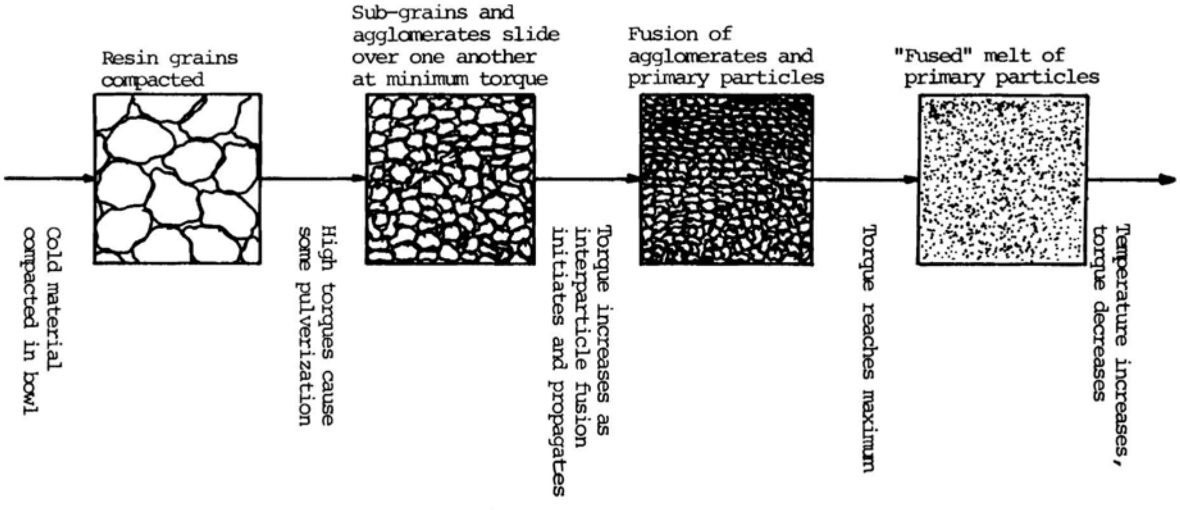
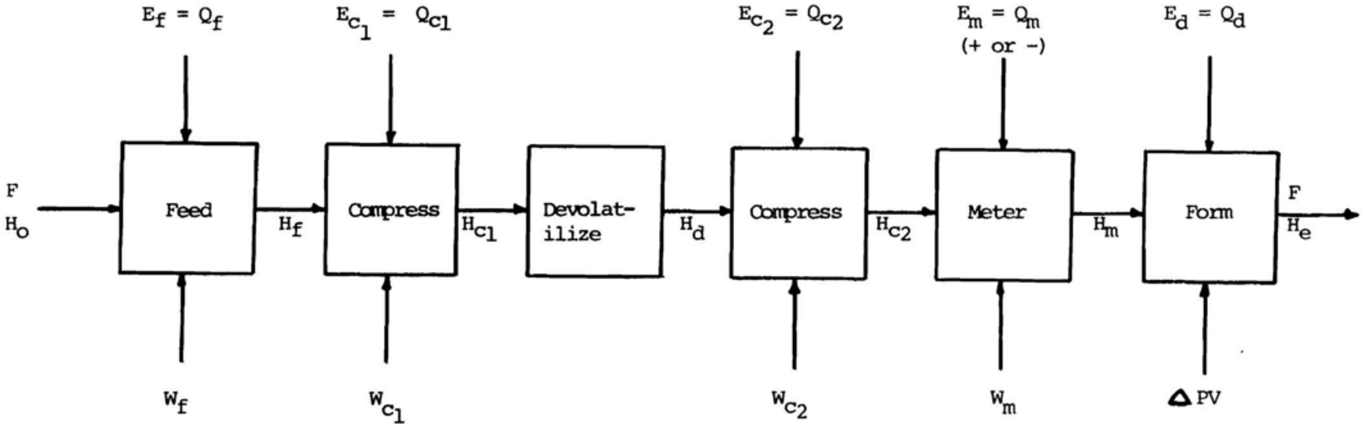


Fig. 6. Rigid PVC fusion mechanism in a torque rheometer.



Where: $E = Q = \int U\,A\,d\,T$
= Rate of heat transfer, BTU/HR
W = Work imparted into material, BTU/HR
= f (equipment, compound, lubricants, feed rate, screw speed, etc. . .)
F = Extruder thruput, LB/HR
$H = F\,C_p\,T + PV$
= Enthalpy of material, BTU/HR

Fig. 7. Simple energy balance for a rigid PVC extruder.

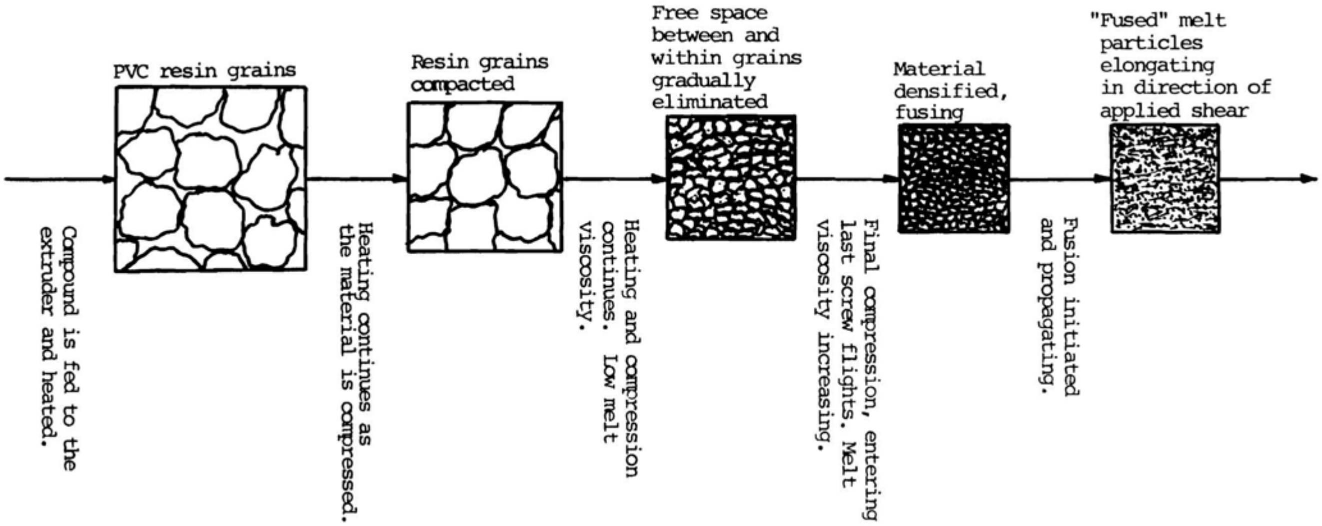


Fig. 8. Rigid PVC fusion mechanism in a twin screw extruder.

involves the elimination of free space between the PVC resin grains, followed by elimination of free space between the primary particle agglomerates, and finally elimination of free space between the primary particles. Interparticle fusion initiates as the melt temperature increases. Finally, as the amount of shear stress increases, the primary particles undergo an elongation in the direction of applied shear. Variations from this mechanism occur with different extruders which impart different amounts of shear into the compound during the early stages of fusion. For example, on many twin screw extruders, the amount of initial compression can be controlled by varying the amount of feed entering the extruder with a starve feeder. Another example is an extruder equipped with mixing zones early in the process prior to compression; this extruder pulverizes the material somewhat whereas an extruder without mixing zones simply compresses the material. The point at which the fusion transitions are initiated and the rate at which they propagate have a substantial effect on extruder loads.

The objective of extrusion is to deliver a melt, at a suitable temperature and degree of fusion, to the die. The state of the melt at the screw tips depends on the characteristics of the material and the amount of energy input, both thermal and frictional, during the extrusion process. The amount of work imparted to the material depends on the levels and types of lubricants used. The amount of heat transferred to the material is a function of barrel and material temperatures. The behavior of a given formulation when slight changes in lubricant levels are made provides insights into the rheological effects and sensitivity of a particular lubricant. For example, suppose an extruder is producing a 60 percent fused melt at the entrance of the die with a melt temperature of 370°F. An increase in paraffinic lubricant level of 0.10 to 0.20 PHR will reduce both the degree of fusion attained and the melt temperature. The additional paraffinic lubricant reduced interparticle friction and hence reduced W_{c1}, W_{c2}, and W_m. If Q_f, Q_{c1}, and Q_{c2} are increased, a similar melt temperature and gelation level can be reattained. The paraffinic lubricant regulates the amount of work imparted into the compound by the extruder through regulating interparticle friction. It is functioning as a fusion delayer. Increases in calcium stearate level, a fusion promoter, has an opposite effect on the thermodynamics of the process. As calcium stearate is increased, interparticle friction increases causing W_{c1}, W_{c2}, and W_m to increase. Thus, Q_f, Q_{c1}, and Q_{c2} need to be decreased if an equal level of fusion is to be maintained at the die entrance.

What level of fusion is desirable at the screw tips? Widely accepted extrusion theory is that the material entering the die should be fully fused and that the die does nothing more than form the finished product. In industrial practice, the die characteristics have a substantial effect on melt fusion during extrusion. As the material is passing through the die, it is at a temperature where fusion and elongation of primary particles occur most easily. Tooling available for PVC pipe production can be quite different and, for a given extruder, require quite different lubricant balances. For example, a low compression die set with relatively slow compression transitions requires that the material entering the die be well and carefully fused. However, a high compression die set, with faster compression transitions, will impart more fusion and particle elongation than a low compression die set. Thus, a higher compression die set fed a moderately fused melt may yield a similar degree of fusion in the end product as a low compression die set fed with a well fused melt. Within the working range of a formulation, the higher compression die set performs better with a dry blend containing a higher level of fusion delaying lubricant whereas the lower compression die set performs well with a higher level of fusion promoting lubricant.

The effects on end product physical properties of compositional changes resulting from small (less than 0.30 PHR) lubricant changes are less significant than the resultant physical property changes due to rheological and morphological changes. Not only do the physical properties of the end product depend on the apparent degree of fusion, they also depend upon the *consistency* of the end product morphology. Some of the causes of variations in melt and finished product morphology include excessive screw and barrel wear, excessive temperature control tolerances, inadequate mixing and compounding, and marginal to poor lubricant balances. As production rates are increased, it becomes more and more important to use consistent quality lubricants in order to maintain a stable extrusion process resulting in consistent finished product morphology and physical properties.

During the last few screw flights and as the material enters the die, the conventional external and internal lubricant terminology becomes more appropriate. Metal release and film formation on the tooling surfaces can have a substantial effect on the quality of the extrudate surfaces, the evenness of melt flow, and end product physical properties. In practice, the film formation characteristics of a lubricant system are very difficult to understand and predict. External lubricants are often thought of as migrating to the surface of the melt. Low molecular weight polyethylene waxes are typically used for their film formation characteristics. After a polyethylene wax is added to or removed from a formulation, it takes days of extrusion before the films which form on the die surfaces equilibrate. The effects of internal lubricants in low shear, relatively low temperature extrusion are also difficult to predict. Internal lubricants are thought of as reducing the viscosity of the melt. However, variations in fusion can cause much greater variations in the viscoelasticity of the melt and pressure drop across a die. A highly fused material can generate twice the pressure drop across a die than a poorly fused melt (11). An effective internal lubricant should yield reductions in melt viscosity at both low and high degrees of fusion.

Improvements in lubrication can lead to significant productivity improvements. *Table 1* outlines the improvements observed when using a new proprietary synthetic lubricant containing calcium stearate *vs.* conventional calcium stearate. As shown in this example, improvements were obtained in output rate, extruder loads, wall control, and product appearance. Extruder output improved approximately 11 percent while output per top screw RPM improved 8.6 percent and output per bottom strew RPM improved 13 percent. This example demonstrates the degree of improvement possible in extrusion through relatively minor changes in the lubricant system.

Table 1. Performance Comparison Between Conventional Calcium Stearate and RHEOLUB 1800

Lubricant Balance	Standard	Improved
165 paraffin wax	1.20	1.00
Calcium stearate	0.60	—
RHEOLUB 1800	—	0.75
Oxidized Polyethylene Wax	0.15	0.15
Screw RPM top/bottom	2175/2800	2225/2750
Motor amps top/bottom	54/72	53.5/69
Output, LB/HR	1440	1600
Wall control, inches	0.241–0.260	0.241–0.255
Appearance, OD & ID	Good	Excellent

Extruder:	A4-125/125 Retrofitted with 127/127 MM barrels and screws
Die:	CT-6100
Product:	Moderately filled 8-inch G/S pipe (cc 12454B)
Feed:	Flood

CONCLUSIONS

1. Rigid PVC lubrication is an important tool for the rigid PVC processor which can have a major impact on the productivity and efficiency of an operation. Improvements in the lubricant system or balance often lead to improvements in product quality and productivity.

2. Industrial optimization of a lubricant system involves balancing compound and equipment characteristics. As a result, PVC lubrication is highly production and situation oriented.

3. The rigid PVC fusion process involves a series of particulate changes from larger to smaller particles followed by fusion and elongation of PVC resin primary particles. The rate at which this process proceeds is influenced by lubrication.

4. It is important to recognize that within the working range of a rigid PVC formulation, some lubricants act to promote fusion and some act to delay fusion. When evaluating internal lubricants, it is important to separate internal lubricating effects from degree of fusion variations.

5. A consistent policy of machine and tooling selection will help insure the rigid PVC processor of maximum product quality and productivity using a minimum number of PVC compounds.

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