Improved Processing and Batch Time Reduction Through Powder Liquid Dispersions

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Abstract

The use of powder liquid dispersions in rubber compounds is well known, and many of the processing, health and safety benefits are well documented. In this paper, the use of powdered concentrates to reduce processing times is discussed.

High-viscosity liquids and resins, as well as high loading of low-viscosity esters and oils, lend themselves to be dispersed on dry carriers and then added to an internal mixer or two-roll mill. In general, these products are dispersions of liquids and semi-solids onto highly absorbent carriers. The advantages of using these products include:

- Elimination of the handling problems associated with high-viscosity liquids and semi-solids by converting them to powder;
- Elimination of the need for heated inventory storage of high-viscosity liquids or non-pouring liquids;
- Reduction in batch preparation time;
- Reduction in equipment clean-up time;
- Improvement in batch incorporation time and raw material dispersion;
- Reduction in the amount of costly materials wasted when viscous liquids or semi-solids stick to the walls and bottoms of drum packages;
- Improvement in package disposability by using fiber drums or paper bags as standard packages.

These benefits can increase productivity and improve profitability. The percent active or the amount of liquid that is absorbed is typically 72 percent.

Introduction

The addition of liquids to rubber compounds leads to problems in handling, incorporation and dispersion. Despite these problems, many compounders incorporate various liquid additives, ranging in form from low viscosity oils to viscous, tacky polymers. In large-volume applications, the installation of storage tanks and metering pumps can offer a solution to these handling problems, but in cases where rapid incorporation of the liquid and good dispersion are required or where the engineering approach is not viable, the use of a powder liquid dispersion (PLD) can eliminate many of the problems normally experienced.

In a PLD, the liquid is absorbed onto a suitable powder (or carrier) to form a product with the appearance of a powder but which contains a high proportion of liquid. The amount of liquid that can be carried depends on the liquid and the carrier used, but 72 percent is the normal loading for many materials so that they can be easily handled without any dust being produced. During mixing, the shear forces developed will break down the structure of the carrier so that the liquid is gradually released into the mix, resulting in rapid dispersion.

Materials

Rubber compounding ingredients vary from ultra-high viscosity elastomers to water-thin liquids with a myriad of powders, resins, accelerators, etc. and with varying degrees of dispersibility. The mixing of all these ingredients together requires specific procedures regarding mixing conditions such as temperature, time, speed of rotors, etc. To ensure quality, many raw materials are modified for enhanced dispersion.

The following is a list of the materials that can be converted to powders.

- Monomeric/Polymer ester plasticizers
- Liquid polymers
  - Nitrile
  - Epichlorohydrin
  - Polybutene
- Process Oils
- Coumarone-Indene Resins
- Pine Tars
- Petrolatums
- Co-agents (monomers)
- Peptizers
- Chlorinated Paraffins
- Accelerators
- Antioxidants
- Process Aids
- Catalysts

Difficult solids can sometimes be handled more conveniently as a PDL. Materials such as TEA, TAC and TAIC are normally supplied as a solid crystalline mass that must be melted in order to be removed from the drums in which they are supplied. Some waxes are so soft that if they are pelletized, they will rapidly agglomerate and form a solid mass. If the materials are converted to a PLD, they will remain relatively stable and will remain as powders even during hot weather. Petrolatums are much easier to handle as a PLD versus the neat form. These products normally present no handling problems. If the mix temperature is low, the time taken to melt and disperse the pellets may be unacceptable. If sufficient time is not taken to achieve a complete melt, fragments of undispersed pellets may survive the mixing process. By turning such materials into PLDs they are incorporated into the mix in a much finer form, and melting and dispersion is much more rapid.

Carriers

The choice of suitable carrier is critical when optimizing the performance of PLDs. The carrier should give acceptable performance in the following areas.

- It must be compatible with the compound and acceptable in the end application.
- It must not react or adversely affect the stability of the liquid component;
- It must be cost effective.
- It must release the liquid under the mixing conditions involved in the process. If a proportion of the liquid is retained in the carrier, the efficiency of the PLD will be reduced.

Amorphous Silica

Precipitated silicas are ideal carriers for use as PLDs. Precipitated silicas are available in a variety of carrying capacities, particle sizes and dispersion characteristics. Silica is a stable material that can be used in a variety of applications, for example, a filler, carrier, thixotropic agent, clarifier and desiccant. These materials have a high degree of porosity and can safely produce PLDs up to 72 percent liquid. These silicas can sometimes react with other chemicals, whereas the use of a calcium silicate can be a simple way of preventing such an interaction. There are grades of calcium silicate that can carry 72 percent by weight of liquid content. The silicates tend to have more reinforcing effect on a rubber compound than the precipitated silicas.

Fumed silicas can be used and have adequate carrying capacity but tend to be dustier and are expensive.

Clays and precipitated calcium carbonate can be used to prepare PLDs with 30 and 40 percent activity, respectively. These tend not to be cost-effective because of the increased amount of product needed for the finished end product.

In some cases, it is possible to use an existing filler from a formulation as a carrier. This does not compromise the formulation and allows for a filler to be wetted, which helps in reducing dusting and aids rapid incorporation of the filler.

Advantages of using PLDs

There are several reasons the use of powdered liquid concentrates can improve production and profitability:

- *Elimination of handling issues associated with high-viscous liquids or semi-solids*

  Many of the plasticizers, resins, waxes and rubber chemicals require heating to reduce their viscosity and make it possible for them to be weighed and dispersed. Due to safety factors, the heating process requires that most of these chemicals be heated slowly to avoid hot spots in the liquid and the possibility of thermal degradation. It will generally take several hours or even several days to reach the acceptable viscosity needed for dispensing. Once a material reaches a temperature at which it can be processed, the equipment used needs to be capable of maintaining this temperature, possibly for long periods.

- *Reduction in clean-up time*

  Unless liquid is metered directly into a suitable internal mixer, containers must be provided for weighing and handling liquids and an appropriate method must be to avoid increased spillage. If disposable containers are used, they will be contaminated by the liquid and will need to be treated as special waste. The cost associated with this type of waste is high and will increase as time goes on. Reusable containers are an alternative but must be kept clean, covered and carefully stored to prevent contamination. PLDs can be without these waste or clean-up issues. An ideal method to eliminate all waste and clean-up is to purchase the PLDs in low-melt bags.

- *Reduction in batch preparation time*

  The elimination of handling drums, pouring out liquids, heating and clean-up will directly affect batch preparation time. Another possible time-saving measure is to purchase the PLDs as preweighs. The combination of using a low-melt bag with a pre-measured amount of PLD will provide large time savings in handling and weighing. The added benefit of using preweighs to improve quality is well documented.

- *Improvement in batch incorporation time and raw material dispersion*

  Mixing liquids of widely divergent viscosities together is a difficult operation. Most of the plasticizers, oils, resins, and liquid polymers are magnitudes lower in viscosity than the elastomers they are being mixed in. An everyday analogy is mixing bread dough. If you try to mix all the oil into the flour at once, you tend to get a lumpy lubricated mixture. To get an even distribution of the oil and have a complete mix, it takes a great deal of work and time. If the oil or liquid is incorporated slowly, the compound will have an even distribution. Table I compares the viscosities of several liquid additives to elastomers.

A PLD subjected to the shear of a mixing process first forms a stiff paste, which quickly incorporates. Further shear reduces the carrier structure, leading to a very gradual release and, accordingly, the uneven softening that occurs with a liquid.

Low-viscosity plasticizers are easy to handle and not normally put onto a silica carrier to improve handling or processing. The problem occurs when adding or injecting these types of materials into an internal mixer; the viscosities of the liquid and the polymer are vastly different, thus causing delay for full incorporation of the plasticizer in the polymer matrix.

In this study, a comparison of DOP both neat and as a PLD was added to a nitrile compound. Table II compares mixing times and energy consumption.

The results show that mixing times can be dramatically reduced. The batch mixing time reduction can improve volume output and lower energy requirements. In an actual factory case, this same data was applied to a similar compound that had a mix time of 210 seconds. By switching to a PLD, the mix time was reduced by 90 seconds. Because this compound was of significant volume, the producer was able to delay the purchase of new mixing equipment.

- *Accurate weighing and improved quality*

Compounders using SPC to control the quality of their products find that replacing a liquid with a PLD improves product consistency. This had traditionally been attributed to the improved accuracy of addition levels achieved with PLDs, but it is probable that the improved dispersion achieved with PLDs also contributes to the reduction in batch-to-batch variation.

Accurately weighing out a powder is a simpler operation than weighing a liquid. Any small spillages of PLD can be swept up easily and will not coat and contaminate scales or other equipment. These properties of clean handling also overcome the problem of allowing for liquid retention in weighing containers. It is almost impossible in practice to completely drain a liquid from a container. Normal practice is to make an allowance for the expected retention when weighing out material, but the amount of material retained can vary considerably. Achieving accurate addition levels becomes difficult, as the operator has to judge when the container has drained sufficiently. Where materials have been heated to reduce the viscosity, some cooling will occur between the materials being weighed and being added to the mix. Viscosity will vary with temperature, and using a standard time to drain the container will not give standard retention of liquid. Scraping out the container will give lower retention figures but can be time-consuming and increases the operator’s chance of coming into contact with the liquid.

TABLE I

Viscosity of Various Liquid Additives to Elastomers

Materials	Viscosity Range, cps at 25°C
Dioctyl Phthalate (DOP)	57
Trioctyl Trimellitate (TOTM)	260
Process Oil	40-5000 SUS at 38°C
Polymeric Plasticizers	1,000–160,000
Liquid Nitrile	20,000–30,000
Polybutene	2,750–35,000
Coumarone – Indene Resin	Solid
Pine Tars	600–solid
Coagents – TAC	Solid
Waxes	Solid

TABLE II

Comparison of Mixing Times of Neat Liquid versus PLD

	DOP	PLD DOP
Plasticizer Level, PHR	**30**	**30**
Rheomix 600, 77 rpm, 93°C		
Maximum Torque, m·g	2080	2400
Compound Temperature, °C	108	111
Maximum Energy, kJ	64	46
Dispersion Time, min.	10.0	7.0