

## RELEASE AGENTS

Release agents are substances that control or eliminate the adhesion between two surfaces. They find considerable use in the adhesive, food, furniture, glass, metal, plastics, rubber, printing, and packaging industries. They expedite many industrial handling and processing operations, particularly of polymers, such as calendering, casting, embossing, extrusion, forming, labeling, laminating, machining, molding, packaging, protecting, and transferring. These release agents are known by a variety of terms descriptive of their effect, including abherents, adhesives, antiblocking agents, antistick agents, external lubricants, surface lubricants, mold-release agents, parting agents, and slip agents. Expectedly, release agents are usually applied directly to surfaces. However, it is sometimes possible to incorporate them into the bulk of the material being treated. Such processing aids are known as internal release agents. They act by migration to the surface and consequently also have an inevitable, internal lubricant effect. Likewise, materials intended as internal lubricants may also exhibit release characteristics, particularly when used at high concentration, if some of the material finds its way to the exterior.

Release agents function by either lessening intermolecular interactions between the two surfaces in contact or preventing such close contact. Thus, they can be low surface-tension materials based on aliphatic hydrocarbon or fluorocarbon groups, or they can be particulate solids. These two categories are by no means mutually exclusive; certain waxes, eg, can exhibit characteristics of both types. The principal classes of materials used are waxes, fatty acid metal soaps, other long-chain alkyl derivatives, polymers, and fluorinated compounds.

### 1. Product Types and Requirements

Release agents are available in a wide variety of forms and are formulated for numerous modes of application. Product types include neat liquids, solutions, powders, flakes, pastes, emulsions, dispersions, sprays, and films. Some are general purpose inert products intended for a broad range of applications, including home consumer uses, whereas others are highly specific, designed to cross-link or react *in situ* with particular substrates. As with most other formulated processing aids, there is a trend away from products containing volatile organic compounds (VOCs) and ozone-depleting substances, and toward water-based and high solids formulations. Users are also switching from the heavier metal soaps such as lead stearate [7428-48-0]. Another trend that helps reduce solvent and carrier use is toward semi permanent release treatments for multiple releases that have to be applied less frequently. Many products serve more than one processing function and contain other additives, such as antioxidants and wetting agents. The composition of these multifunctional and reactive products is usually proprietary information.

Suppliers of release agents vary from large, basic polymer producers to small, regional formulators and distributors. Examples from among the hundreds of such suppliers include the following:

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Axel Plastics Research Laboratories, Inc.  
Barlocher GmbH  
Chem-Pak, Inc.  
Dexter Adhesive and Coating Systems  
Dicronite Dry Lube  
Dow Corning Corp.  
DuPont Coating and Release Systems  
Endura Coatings, Inc.  
Ferro Corp.  
Th. Goldschmidt AG  
Kluber Chemie KG  
Mann Formulated Products, Inc.  
Price-Driscoll Corp.  
Releasomers, Inc.  
Rhodia, Inc.  
Witco Corp.

Many more suppliers and information about their product line can be obtained from compilations such as the *Modern Plastics World Encyclopedia* (1) and the *Thomas Register of American Manufacturers* (2). Obviously, the choice will depend on the processing conditions involved and the nature of the contacting substrates (3). In addition to ease of release, other important requirements are minimal buildup of residues on mold substrate, minimal effect on the molded article, adequate film forming ability, compatibility with other operational conditions and procedures, health and safety requirements, and cost.

The prevention of residue buildup in polymer processing from either the mold release agent or the molding compound is a common product shortcoming (4). Should it occur, ease of cleanability of the mold surface becomes important. Undesirable effects on molded article surfaces include blistering, streaking, discoloration, stress cracking, and swelling. Film-forming ability is critical in casting and molding operations where melt flow might displace the release agent. Cohesive strength and substantivity to the mold can be enhanced by incorporation of polar groups but this may have a detrimental effect on release performance. Time is often a key operational condition. For example, water or solvent carriers must fully evaporate before a mold is filled. This can be critical in automated processes and limits release agent choice. Similarly, elevated temperature applications require good thermal and oxidative stability to prevent formation of polar, adhesive like groups. Health and safety concerns are use specific; foodstuffs and drugs processing and packaging requiring the most stringent consideration. Cost effectiveness, the number of acceptable releases per unit cost, is the key cost-related requirement.

These criteria guide product selection, but trials under production conditions are prudent. Some standard test methods are available that closely simulate actual end-use conditions. For example, ASTM D3354-89 (5) quantifies the degree of blocking, i.e. unwanted adhesion, between plastic layers. There are also standard test methods for determining coefficients of friction (D1894-95 and D3028-95) and surface gloss and haze (D2475-90 and D1003-95) that can reflect release agent effects on substrate surfaces. The impact of release additives on bulk properties must also be considered, particularly with integral additives to plastics. For example, partial solubility usually confers some plasticizing effect that may improve impact strength but could reduce heat distortion temperature. Some release additives such as metallic soaps have antioxidant and heat-stabilizer benefits that are exploited in multipurpose formulations.

**Table 1. Chemical Classification of Release Agents**

Chemical class	Chemical subdivisions	Specific examples	CAS Registration Number
waxes	petroleum waxes	paraffin wax	[8002-74-2]
	vegetable waxes	carnauba wax	[8015-86-9]
	animal waxes	lanolin	[8006-54-0]
	synthetic waxes	polyethylene wax	[802-88-4]
fatty acid metal soaps	metal stearates	magnesium stearate	[557-04-0]
other long-chain alkyl derivatives	other	calcium ricinoleate	[6865-33-4]
	fatty esters	diethylene glycol monostearate	[106-11-6]
	fatty amides and amines	ethylenebis(stearamide)	[110-30-5]
polymers	fatty acids and alcohols	oleic acid	[112-80-1]
	polyolefins	polypropylene	[9003-07-0]
	silicones	poly(dimethylsiloxane)	[9016-00-6]
	fluoropolymers	polytetrafluoroethylene	[9002-84-0]
	natural polymers	cellophane	[9005-81-6]
	other	poly(vinyl alcohol)	[9002-89-5]
fluorinated compounds	fluorinated fatty acids	perfluorolauric acid	[307-55-1]
inorganic materials	silicates	talc	[14807-96-6]
	clays	kaolin	[1332-58-7]
	other	graphite	[7782-42-5]

## 2. Classification of Release Agents

A classification by chemical type is given in Table 1. It is neither rigorous nor complete and some materials could appear in more than one of these classifications. Polyethylene waxes [9002-88-4], eg, could be considered both as synthetic waxes and polyolefins. The broad classes of release materials are listed in the chemical class column, and the principal types in the chemical subdivision column, with a single example in the specific examples column. No attempt has been made to incorporate actual commercial products as many are mixtures and some are of proprietary composition. For example, metallic soaps are often used in combination with hydrocarbon waxes to produce finely dispersed suspensions. Many products also contain formulating aids such as solvents, emulsifiers, and biocides.

## 3. Mechanism of Release

Because release agents are used to reduce the adhesion between materials, it is self-evident that the mechanisms of adhesion and the properties that affect it are the key to understanding release phenomena. These main mechanisms are chemical reaction across the interface, interdiffusion, electrostatic attraction, surface energetics and wettability, and mechanical interlocking. Important surface and interfacial properties include surface topography, surface composition, surface energy and its effect on substrate and adhesive wettability, and the thermodynamic work of adhesion. Key bulk properties of the thin-film interfacial phase include its ability to set to a cohesive solid after wetting the substrate and its viscoelastic response to deformation controlled by factors such as degree of crystallinity, molecular weight and distribution, number of cross-links, and the presence of fillers. The absence of weak boundary layers can also be crucial to developing good adhesion between phases.

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Inversion of these arguments indicates that release agents should exhibit several of the following features: (1) act as a barrier to mechanical interlocking; (2) prevent interdiffusion; (3) exhibit poor adsorption and be unreactive with at least one of the contact phases; (4) have low surface tension, resulting in poor wettability, ie, negative spreading coefficient, of the release substrate by the adhesive; (5) low thermodynamic work of adhesion; (6) low intermolecular forces across the interface, eg, an absence of electrostatic or polar attractions; (7) display nonsetting or low cohesive interactions within the release phase; and (8) provide a weak boundary layer.

Many of these features are interrelated. Finely divided solids such as talc [14807-96-6] are excellent barriers to mechanical interlocking and interdiffusion. They also reduce the area of contact over which short-range intermolecular forces can interact. Because compatibility of different polymers is the exception rather than the rule, preformed sheets of a different polymer usually prevent interdiffusion and are an effective way of controlling adhesion, provided no additional strong interfacial interactions are thereby introduced. Surface tension and thermodynamic work of adhesion are interrelated, as shown in equations 1–3, (6) and are a direct consequence of the intermolecular forces that also control adsorption and chemical reactivity.

The work of adhesion,  $W_A$ , is the change in energy per unit surface area when two phases come into contact, as shown in equation 1 where  $\sigma_1$  and  $\sigma_2$  are the surface energy of each phase and  $\sigma_{12}$  the interfacial energy between them.

$$W_A = \sigma_1 + \sigma_2 - \sigma_{12} \quad (1)$$

. If the adhesive is phase 1 and the release coating is phase 2, then the spreading coefficient,  $S$ , of 1 on 2 is as given in equation 2.

$$S = \sigma_2 - \sigma_1 - \sigma_{12} \quad (2)$$

Because the work of cohesion,  $W_C$ , of the adhesive is  $2\sigma_1$ , equation 3 follows.

$$W_A - W_C = S \quad (3)$$

Thus when the forces of adhesion are less than those of cohesion, ie, when  $W_A < W_C$ , the spreading coefficient is negative and failure will be at the interface between the adhesive and release coating, the desired situation in a release coating application. Conversely, when the spreading coefficient is positive, undesirable cohesive failure in the adhesive or release coating will be the case. These simple, fundamental equations demonstrate the clear requirement that the release coating surface energy be lower than that of the adhesive. Table (2) lists the surface energy of some common release substrates.

There are three broad types of intermolecular forces of adhesion and cohesion (13): quantum mechanical forces, pure electrostatic forces, and polarization forces. Quantum mechanical forces account for covalent bonding. Pure electrostatic interactions include Coulomb forces between charged ions, permanent dipoles, and quadrupoles. Polarization forces arise from dipole moments induced by the electric fields of nearby charges and other permanent and induced dipoles. Ideally, the forces involved in the interaction at a release interface must be the weakest possible. These are the polarization forces known as London or dispersion forces that arise from interactions of temporary dipoles caused by fluctuations in electron density. They are common to all matter and their energies range from 0.1 to 40 kJ/mol. Solid surfaces with the lowest dispersion force interactions are those comprised of aliphatic hydrocarbons and fluorocarbons and that is why such materials dominate the classification table (Table 1) and the surface energy table (Table 2).

Aliphatic fluorocarbons are the lowest surface-energy solids known. Among the hydrocarbons, the lowest surface energy values are found for surfaces comprising closely packed methyl groups. Zisman (14) has shown from critical surface tension of wetting studies that the order of increasing surface energy of fluorocarbon and

**Table 2. Surface Tensions of Various Release Substrates**

Substrate	CAS Registry Number	Surface tension, <sup>a</sup> mN/m(=dyn/cm)	Reference
carnauba wax	[8015-86-9]	38 <sup>b</sup>	7
paraffin wax	[8002-74-2]	23	8
octadecylamine monolayer	[124-30-1]	22	9
stearic acid monolayer	[57-11-4]	21	9
polyethylene	[9002-88-4]	33	10
polypropylene	[9003-07-0]	29	10
polydimethylsiloxane	[9016-00-6]	24	10
polymethylnonafluorohexylsiloxane	[115287-18-8]	16.3	10
poly(vinyl fluoride)	[24981-14-4]	28	10
poly(vinylidene fluoride)	[24937-79-9]	25	10
polytetrafluoroethylene	[9002-84-0]	18.3	10
10-monohydroperfluoroundecanoic acid monolayer	[1765-48-6]	15	11
poly(1,1-dihydroperfluorooctyl methacrylate)	[29014-57-1]	10.6	10
<i>n</i> -perfluoroeicosane	[37589-57-4]	6.7 <sup>c</sup>	12
perfluorolauric acid monolayer	[37589-57-4]	6	11

<sup>a</sup>Critical surface tension of wetting, *n*-alkane test liquids only, unless otherwise indicated.

<sup>b</sup>Extrapolated value from higher temperature measurements on liquid material.

<sup>c</sup>Critical surface tension of wetting, mixed series of test liquids.

hydrocarbon constituent groups is  $\text{CF}_3 - < - \text{CF}_2 - < \text{CH}_3 < - \text{CH}_2 -$ . Organic waxes and other long-chain alkyl derivatives achieve a methyl-rich surface by alignment of the alkyl chains. A related surface can be obtained with plentiful methyl groups along a polymer chain as in the case of poly(dimethylsiloxane) [9016-00-6]. Such is the extreme localization of these atomic force fields, that there is little direct effect on surface energy from the underlying partially polar siloxane backbone. Because of this very short, nanoscale range of operation of dispersion forces, release can also be effected by minimizing the degree of contact between surfaces using finely divided solids. Some, such as microcrystalline wax [64742-42-3] and hydrophobic silica [7631-86-9], combine the two effects in what is sometimes referred to as a microscopic ball bearing mechanism. Recent studies on the fundamentals of release have also pointed to the importance of a low glass transition temperature as well as low surface energy. See, eg, the studies of Newby and Chaudhury (15) on fundamentals of release from viscoelastic adhesives. More detailed publications on specific mechanistic aspects of release agents are available, eg, metallic stearates (16), fatty amides (17), application of fluorochemicals to textiles (18), release of polyurethanes (19), and silicone-coated release papers (20).

#### 4. Economic Aspects

The low surface energy and inertness of the materials used as release agents are exploited in numerous other coating applications and processing aid operations, such as water repellents, polishes, defoamers, and pigment and filler treatments. Consequently, it is difficult to obtain unambiguous economic information regarding release agent consumption. The 1997 *Encyclopedia* estimate quoted an earlier 1992 figure for worldwide consumption of lubricant and mold-release plastics additives of  $130 - 140 \times 10^3$  metric tons. The United States, Western Europe, and the rest of the world accounted for one-third each of this activity, which is still a good estimate, as the small growth in new markets is balanced by the trend toward semipermanent mold-release compounds.

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Estimates can also be made for certain product types in applications that are critically dependent on release agents. For example, silicone release coatings for pressure-sensitive adhesives account for  $\sim \$100 \times 10^6$  in the United States, with the rest of the world consuming a similar amount. Growth is limited in many of these release applications with price increases largely offset by production and use efficiencies. Release agent product prices cover a very broad range from a few dollars per kilogram for simple solvent dispersions of stearates and paraffin waxes up to \$100/kg for some complex, reactive, proprietary compositions. They may sometimes be the only alternative, but they are often cost effective even when less expensive materials can be used, because of the lower use level or less frequent application rate required. A release agent should be selected not on the basis of unit cost, but for its impact on overall processing and finishing costs.

### 5. Health and Safety Factors

Most general purpose release agents have been developed for this market in part because of their low toxicity and chemical inertness and do not usually present health and safety problems. Some of the solvent dispersions require appropriate care in handling volatile solvents, with some suppliers offering water-based alternatives. Some of the solids, particularly finely divided, hydrophobic solids, can also present inhalation problems. Some of the metallic soaps are toxic, although there is a trend away from the heavier, more toxic metals such as lead. The reactive type of release coating with monomers, prepolymers, and catalysts can require specialized handling procedures. The potential user with health and safety questions is advised to consult the manufacturer directly.

Principal health and safety concerns involve contact with foodstuffs and drugs. U. S. Government regulations governing the use of additives such as release agents are listed in the *Code of Federal Regulations*. Title 21 contains the rules established by the U. S. Food and Drug Administration. Part 175 deals with indirect food additives, ie, adhesives and components of coatings; part 175.300 specifically covers resinous and polymeric materials for use as components of coatings. This regulation contains a section on release agents that are the basic polymer of the coating, including polyethylene wax, poly(tetrafluoroethylene), and silicones, as well as a section on surface lubricants, including glyceryl monostearate [31566-31-1]. Regulations are subject to change, and the *Code of Federal Regulations* is revised at least annually and also updated in the daily issues of the *Federal Register*. Details of regulatory practice differ between countries and a multitude of regulations exist worldwide. Local health and safety information on a particular product is best obtained directly from the formulator or manufacturer. Their skill and experience is the best defense against hazards associated with the use of release agents.

## 6. Uses

### 6.1. Metal and Glass

Although one might expect that high thermal stability would be a prerequisite for release agent use with molten metals and glasses, it is not necessarily the case. Mineral oils are widely used in molding and shaping glass, despite the smoke and residue buildup entailed. Heat-resistant, graphite-containing resins are also used. Phenolic resins are used in this application and also in mold fabrication for metal castings.

In this process, sand and resin are mulled together and applied to a pattern and hardened to give a mold. Release agents prevent adhesion of the resin binder to the pattern, enabling the mold to be cleanly separated. They also improve the mold release properties after the casting of the metal. Internal mold release agents such as calcium stearate [1592-23-0] have other beneficial effects, such as reduced agglomeration of the resin-coated particles and lower muller power requirements. External release agents such as aqueous silicone emulsions or wax dispersions are used both in the mold making and casting steps. Similar release agents are used in

a variety of other metal-forming operations, including the injection molding of low melting aluminum alloys, drawing of ferrous metals, and hobbing and stamping with application to both the tool and product surfaces.

## 6.2. Rubber and Plastics

Release agents are widely used to achieve release of polymers and release from polymers. They are useful in many polymer-processing applications, such as extrusion, calendering, molding, and embossing. By preventing the polymer from sticking and building up on process equipment, rejects are reduced or eliminated. In subsequent machining, packaging, and labeling operations, adherents allow faster, more continuous handling. Release from polymer applications varies from the familiar example of fluorocarbon-treated cookware to the use of shrink films of poly(tetrafluoroethylene) [9002-84-0] or polyethylene [9002-88-4] over rollers in the ink, printing, polymer processing, and coating industries. A significant aspect of such applications is antideposition coatings to reduce accumulation of pollutants and other undesirable matter. Examples include nonthrombogenic surfaces in biomedical uses, low soiling materials for solar-energy collectors, and soil-release agents in powdered detergents. Antiredeposition agents and soil-release polymers have become important additives to today's laundry products (21).

In the extrusion of thermoplastic polymer powder or granules, release agents aid in the flow and keep the finished product from sticking when stacked. They can also reduce the possibility of thermal breakdown by reducing energy requirements and cycle times. Production of granules of particularly adhesive polymers such as polystyrene [9003-53-6] would be impossible without such aids. In the mixing, sheeting, and calendering of natural rubber [9006-04-6] release agents prevent sticking to rollers, thereby maintaining continuous operation. Waxes, metal stearates, and silicones are widely used; solid adherents are popular for dusting finished, unfilled products.

Molding operations such as the manufacture of automobile seat foams and reaction injection molding (RIM) and blow molding techniques consume considerable quantities of release agents. Many polymers are molded, but polyurethanes present a particular challenge, owing to the likelihood of adhesive bonding of isocyanates to the mold. Polymer films are often used to allow castings to separate from the mold. A specific example is the use of regenerated cellulose (cellophane [9005-81-6]) films in the manufacture of corrugated, glass-reinforced polyester building panels. The molds can also be made from inherently releasing polymers such as silicones.

## 6.3. Adhesive Transfer Processes

There are several important material-transfer applications involving polymer products that are so critically dependent on controlled adhesion that they merit separate mention from general polymer release problems. They include hot stamping foils, release coatings for pressure-sensitive adhesive products, photocopier materials, transfer coatings, and transfer printing of textiles.

Hot stamping foils are a type of composite, decorative laminate that uses a polymer carrier film, either polyester or cellophane, together with an external release agent, usually a wax, to transfer coatings to surfaces to be decorated. The transferred coating is rapidly bonded using heat and pressure. Particular attention must be paid to foil detail and surface area, nature of the transferred coating, and avoidance of premature release during slitting and winding of the foils. Some foils are metallized by vacuum deposition; hence there must be no volatiles or migration to interfere with subsequent adhesion on hot stamping.

Silicon products dominate the pressure-sensitive adhesive (PSA) release liner market, but Werner-type chromium complexes such as stearato chromium chloride [12768-56-8] are also used. Various substrates are used, including coated Kraft papers and polyethylene or polyester films. Silicone coatings that cross-link to form a film are used in various forms such as solvent and solventless dispersions, and emulsions. Technical requirements include good release, no contamination of the adhesive, adequate anchorage to the substrate,

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no blocking in rolls, good solvent holdout with solvent-based adhesives, and good thermal and dimensional stability. Poly(dimethylsiloxane)-based products are used for organic PSA release; fluorosilicones, such as polymethylnonafluorohexylsiloxane [115287-18-8] are needed for silicone PSAs.

Transfer of toner material between rollers and eventually onto the paper is a demanding process. Because speed is of the essence, a combination of temperature and pressure is used to ensure adequate flow of the thermoplastic toner when fusing the image. Temperatures close to 200°C are used, requiring good thermal stability of both the rollers and the release agents. To prevent adhesion of melted toner to the heated roller, its outer layer, at least, is usually a silicone or fluorocarbon material. Often a release agent or fuser oil is also applied to the fuser roll to ensure adequate release. Silicones are widely used on account of their excellent thermal and chemical stability. Another approach is to incorporate the release agent in the toner. Polyethylene and polypropylene [9003-07-0] waxes are dispersed in the toner and migrate to the surface during the fusing step.

Transfer coating is a combination of casting and coating used to create composite materials such as simulated suede leather. A polyurethane film is cast on a release sheet and dried; a second solution is cast on the dried film and then laminated to a suitable fabric when still wet. The release paper is then stripped away when the material has dried. Flexographic printing directly onto fabrics is beset by shrinkage and register problems that are considerably eased by transfer printing from a release sheet of adherent-treated paper. This release sheet is printed on the release side by the direct gravure method, dried, and rewound. Direct gravure offers precision and color possibilities not otherwise obtainable. The roll of preprinted release is then forwarded to the fabric converters for unwinding and pressure–heat lamination of the preprint to the fabric.

### 6.4. Furniture

The furniture industry uses a lot of auxiliary release agents in the general gluing and veneering of wood and wood-based materials, as well as in a variety of coating processes. For example, when applying paper impregnated with phenolic and melamine resins for wood surface finishes, the reverse side of the sheet must be coated with a release agent to prevent sticking to the press surface. Mineral oils, paraffin waxes, fatty esters, and specialized paintable silicones are all used for this purpose. Silicone rubber molds are also utilized in the furniture industry; they generally have adequate release properties and require no additional release agents.

### 6.5. Food

Household food uses of release materials such as polyolefin refrigerator ice trays, other foodstuff molds, wax paper, release-coated pans and bake ware, and nonstick cooking sprays are very familiar. One of the oldest methods is the use of flour to release dough from preparation surfaces. Other natural adherents include confectioners' sugar, rice flour, and rice paper. The use of adherent surfaces in the food industry not only decreases product loss, but also eases equipment cleaning. Semi-permanent release coatings based on silicone resins and polytetrafluoroethylene can last for months whereas wax coatings are removed after each batch of cooking during cleaning and reapplied prior to the next use. Vegetable and animal fats applied pure or as emulsions are used in this way in the bakery industry. Bakery liners are also employed, and can often be reused several times. Glassine and parchment papers treated with chromium complexes are examples. Note that the trivalent chromium complexes used do not share the hexavalent chromium health hazard.

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