CONCENTRATED LAUNDRY POWDER DETERGENT

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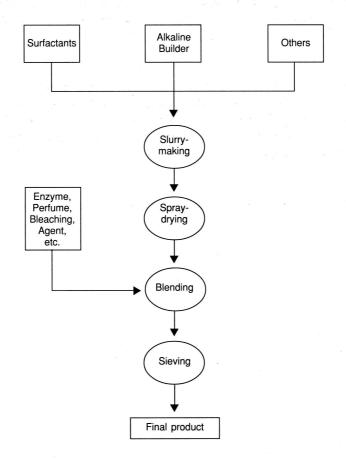
RINGKASAN: Perubahan terkini di dalam teknologi produk serbuk pencuci pakaian adalah pertukaran daripada serbuk pencuci biasa kepada serbuk pencuci pekat (padat). Artikel ini mengulas mengenai jenis produk pencuci pakaian dan pasarannya, kandungan bahan di dalam produk serbuk pencuci pekat dan cara proses pengeluarannya.

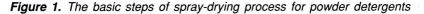
ABSTRACT: The most recent change in detergent powder technology has been the move from conventional to concentrated (compact) laundry powder detergent. This article reviews the types of laundry detergent in existence today, their consumption, typical composition and the process to manufacture concentrated laundry powder detergent.

KEYWORDS: Laundry powder detergent, conventional, concentrated (compact).

INTRODUCTION

Since the early 1960's, detergent powders were produced by spray-drying process. The basic steps in the manufacture of powder detergent using this process are represented in Figure 1. In this process, detergent ingredients such as surfactants and builders are mixed with water to form slurry having a water content of 35% to 50% by weight. The slurry thus obtained is sprayed after heating, into a heated space in a spray dryer to form hollow beads having a water content of 5% to 10% by weight and bulk density of 300-450 g/l. This produces granular detergent having an excellent solubility. However, since 30% to 40% by weight of water must be removed in the drying step, an extremely large amount heat energy is consumed. Furthermore, since the granules obtained by spray-drying have low bulk density, the packaging volume becomes disadvantageously large. Laundry powder detergent produced using spray-drying process system is represented in Figure 2 (Falbe, 1987).





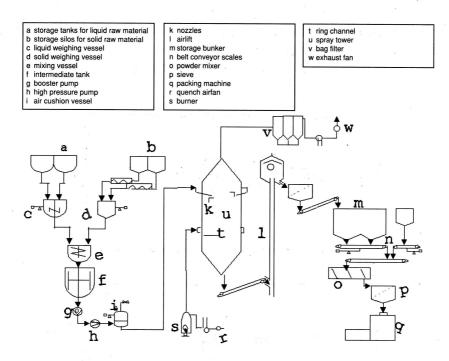


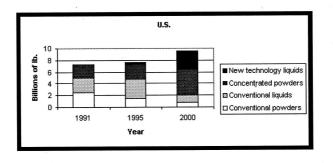
Figure 2. Flow diagram of a spray-drying process for powder detergents

In the last few years, due to changes in washing habits and spurred by consumer concerns for the environment, detergent makers around the world have reformulated their products. Recently, considerable interest has been shown within the detergent industry, as well as among consumers, in concentrated (compact) detergent powders. The advantages of these products are: (i) smaller containers or packs provide easier handling to the consumers, (ii) savings in storage and transport costs, (iii) smaller packs create shelf space for stacking more packs, and (iv) less packing material will result in less waste to the environment.

MARKET

The trend on the market of laundry detergent in U.S., Europe, and Japan are shown in Figure 3 (Thayer, 1993). Concentrated detergents captured a 29% share of the U.S. market in the early 1990's. Most companies anticipate that concentrated laundry detergent will continue to be a major trend, having now been fully introduced nation wide in the U.S. European manufacturers introduced the concentrated laundry detergent in 1990. These products have a weaker market penetration, about 7% in 1993. However, some producers of detergent raw materials estimate that the market share held by concentrates in Europe is as high as 30%, but differs on a country-by-country basis. In U.S. and European markets,

concentrates are expected to continue to gain market share in the next decade while conventional powders and liquids give way to new technology liquid laundry detergents such as structured liquid detergents and non-aqueous liquid detergents (Thayer, 1993).



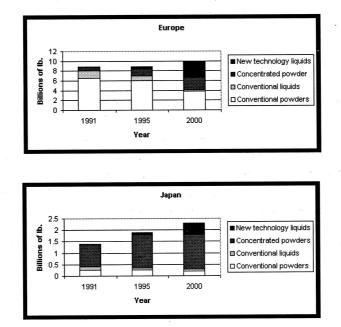


Figure 3. Laundry detergent market trend in U.S., Europe and Japan

During the 1970's, the Japanese detergent industry had introduced energy and resource saving concentrated detergent whose dosage is half that of conventional detergents. However, the market disappeared by the early 1980's because energy saving alone was no longer enough to satisfy consumers. A compact, heavy-duty powdered detergent brought about by newly developed ingredients and having only one-fourth the volume of conventional detergent, were introduced in 1987. By 1992, concentrated powdered detergent captured 80% of the Japanese

market. The trend on the market of laundry detergent in Asian and Pacific countries is difficult to estimate because comprehensive statistical information is unavailable (Croy, 1994).

INGREDIENTS AND FUNCTIONS

The key ingredients in typical laundry detergent products as well as their principal functions are discussed in the following sections:

Surfactants

As a result of their unusual molecular structure, surfactants are able to provide the most important performance attributes of a laundry product by (i) reducing the surface tension of water, (ii) improving wetting conditions, (iii) removing greasy soil, therefore providing detergency, and (iv) foaming.

The primary surfactants for powder formulations are both anionic and nonionic types. Linear alkylbenzene sulfonates (LAS) are still the most commonly used anionic surfactants worldwide, and it is generally agreed that when used correctly, they have little adverse effect on the ecosystem. They have good all-round performance characteristics for a wide variety of fabrics and wash conditions. In Europe, other anionics are used to a lesser extent, and these include paraffin (alkyl) sulfonate (SAS), alpha olefin sulfonates (AOS) and synthetic alcohol ethoxy sulphates (FES). Powder detergent using alpha sulfonated fatty methyl esters, are also available in Japan and Germany (Falbe, 1987).

Products using renewable anionics are predicted to become future rivals to the conventional petroleum-based products. These would undoubtedly include fatty alcohol sulphates (FAS) and fatty alcohol ethoxysulphates (FES), both derived from natural feedstocks such as palm, palm kernel and coconut oils (Whalley, 1995).

Due to the development of new textiles, the lowering of wash temperature, and the replacement of phosphates in detergents, nonionic surfactants have achieved in recent years ever increasing importance. The critical micelle concentration of nonionic surfactants compared to that of anionics is often lower by a factor of 10. Therefore, the nonionic surfactants already show at relatively low concentration, not only excellent soil removal but also good dispersing capability and distinctive soil anti-redeposition effects. Such nonionic surfactants are frequently primarily alcohol ethyloxylates (AEO) containing 3 - 12 mol of ethylene oxide. The degree of ethoxylation is not too critical, provided the nonionic is maintained in solution at higher washing temperatures. The use of nonionic alkylphenol ethoxylates (APEO) in laundry products has been voluntarily stopped or restricted in certain European countries, because of concern over their comparatively slow biodegradation and the ecotoxicity of the degradation intermediates (Whalley, 1995a).

Builders

Detergent powders contain builders to assist surfactant performance and often include sodium carbonate, sodium silicate, and sodium tripolyphosphate. Builders are used to avoid formation of insoluble surfactant salts owing to water hardness and to provide soil emulsification and dispersion and alkalinity/buffering. Sodium tripolyphosphate is probably the most efficient available inorganic builder, but in mid 80s, phosphates were virtually eliminated from domestic detergents in Germany, Italy, Switzerland and the Netherlands. This was due to the perceived relationship between detergent phosphates in waste water and problems of nutrient enrichment in freshwater lakes and rivers. In other countries, voluntary agreements have been reached between the authorities and manufacturers. The enforcement of legal restriction on the phosphate content in detergent was facilitated by the development of zeolite A, which made available a suitable phosphate substitute. This removes calcium and to a lesser extent, magnesium from the wash water by ion-exchange. But unlike sodium tripolyphosphate, zeolites do not act as pH buffers or anti-redeposition agents, and so additional co-builders such as polycarboxylates (PCA) have to be used. PCA have both water softening and soil anti-redeposition properties. Newer zeolite types and layered silicates, especially the socalled disilicates, are still being researched (Coffey and Gudowicz, 1990). Sodium silicates soften water by forming finely dispersed precipitates that can subsequently be rinsed away very easily. In addition, silicate provides alkalinity and corrosion inhibition. Sodium carbonate is used not only as an absorbent agent but also as an alkaline builder. This carbonate is a cheap source of alkalinity in detergent builder. Citrate is used in some products as a builder and dispersant, however its cost and limited sequestration ability have confined it to a minor role (Whalley, 1995b).

Fillers

Inorganic compounds like sodium sulphate are commonly required as filling compounds for powder detergents. The formulation of concentrated detergents minimizes the use of sodium sulphate.

Bleach System

Not all types of soil occuring on household fabrics can be removed by simple washing. In order to achieve a satisfactory cleaning effect hydrated sodium perborate has been used as a bleaching agent in European powders for many years, and is available in forms suitable for conventional and compact detergents. Low washing temperature in the range 40-50°C require the use of bleach activator, which can react with the perborate in solution to form the active bleach component, peracetic acid. The most widely used activator today is

6

tetraacetyl ethylenediamine (TAED), although nonanoyloxybenzenesulfonate (NOBS) is used in the US (Milne, 1998).

Other ingredients

These include perfume to improve marketing as well as other minor ingredients like fluorescent whitening agents (TWA), anti-redeposition agents (carboxymethylcellulose: CMC) and enzymes. Some ingredients, e.g., enzymes, command a higher price than the rest of the ingredients and therefore their use in detergents, although beneficial to performance, will depend on the economics of the geographic area where the product is being marketed.

TYPICAL FORMULATIONS

The variety of potential combinations of ingredients reflects the diversity of formulations existing today. Due to wide differences in washing habits and washing machine types, the formulation structure for conventional laundry powders and concentrated powders vary significantly between the various regions of the globe. These differences are illustrated in Table 1 and 2 (Herman De Groot *et al.*, 1995). In response to eutrophication problems, some countries instituted limitations on the allowable maximum levels of phosphates while others have instituted total bans on phosphate. Conventional laundry powders contain about 10% surfactant, while concentrates contain 20% or even higher with corresponding reduction in the solid filler content. This increases, together with the corresponding increase in quantities of other ingredients, such as bleach and activator, anti-redeposition agents, fluorescent whitening agents, etc.

Component (%)		Conventional Powders from			
		Europe	USA	Far-East	Japan
Surfactants	Anionic	8-12	10-20	15-25	20-25
	Non-ionic	3-5	0-5	0-5	0-3
Builders +	Co-Builders	35-40	25-45	35-50	30-35
Bleaches +	Activators	15-25	0-5	0-10	0-10
Fillers		15-25	20-30	5-30	30-35
Additives		2-7	0-3	0-3	0-5

Table 1. Formulation structure of conven	tional powders
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Component (%)		Concentrated Powders from			
		Europe	USA	Far-East	Japan
Surfactants	Anionic	5-12	15-25	15-30	30-35
	Non-ionic	6-10	5-10	8-12	3-5
Builders +	Co-Builders	25-40	40-60	20-30	40-45
Bleaches +	Activators	18-25	0-5	5-15	5-10
Fillers	·	5-12	0-5	0-10	0-3
Additives		3-10	1-3	3-8	3-6

Table 2. Formulation structure of concentrated powder	Table 2.	Formulation	structure o	f concentrated	powders
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MANUFACTURING PROCESS

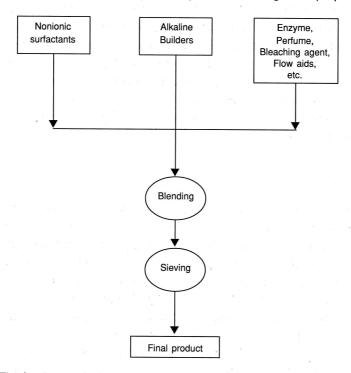
For the concentration of powdered detergents and to achieve smaller packs, in principle the following possibilities exists (i) using more active components, (ii) avoiding activity losses during the manufacture and storage, (iii) minimizing the amount of or avoiding all non-functional ingredients used in the manufacturing process, and (iv) minimizing the amount of air and moisture in the product as well as in the packet.

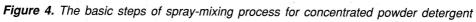
Generally, there are two main types of process to manufacture high bulk density concentrated laundry powder detergent. In spray-mixing process, the various components are dry-mixed and agglomerated with liquids, e.g. nonionic surfactant. The second process involves granulation of detergent components.

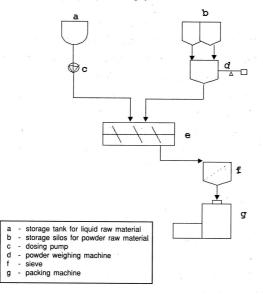
Spray-Mixing Process

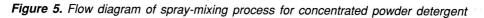
The basic steps in the manufacture of powder detergent using spray-mixing process are represented in the diagram in Figure 4. In this process the liquid nonionic surfactants are frequently sprayed directly onto a powder mixture that is being distributed by air or mechanical means in the mixer. Rotary Mixer, type V-Blender, which has an internal agitator for breaking up lumps and liquid spraying system, is suitable. An advantage of this process is that nonionic surfactants can be incorporated up to a level of 20%. The powder may be coated in one of two ways; (i) the powder raw materials are mixed together before being sprayed with the liquid surfactant, or (ii) lightweight carbonate, phosphate, or silicate is sprayed with the liquid surfactant to form a very bulky, free-flowing powder, which is then mixed with the rest of the powder components (BASF Technical Information, 1987). The addition of inorganic particulate as flow aids, such as colloidal silica, precipitated silica, magnesium silicate, and zeolites, does frequently improve the tackiness of these products, especially those with high contents of nonionic surfactants (Staley, 1997). Essential oils or perfume may be mixed with the powder materials

or sprayed separately onto the coated powder. Concentrated laundry powder detergent is produced using spray-mixing process system represented in Figure 5 (Ropien, 1993).









In this process, the most important factor which governs the bulk density of a detergent powder is the bulk density of the starting materials. Increasing the content of the relatively dense sodium sulphate can increase the bulk density of a spray-mixed powder, but this does not contribute to the detergency of the powder. The detergent prepared by this process are often fine powders which can 'smoke' or cause sneezing and eye irritation when they are poured out of a container during use. Therefore, attempts have been made to make free flowing and dust-free particulate detergent composition with increased concentration of active ingredients and increased bulk densities. It is achieved by developing the granulated detergent technology. Generally, there are two types of granulated detergent technology, namely, agitation granulation and disintegration granulation.

Agitation Granulation

In this process, the traditional spray-drying is used to make highly water-soluble hollow detergent particles. High-density powders can be obtained from spray-dried powders by the spray-addition of liquid nonionic surfactant and subsequent mechanical agglomeration. The basic steps in the manufacture of powder detergent using agitation granulation process are represented in the diagram in Figure 6. In this process, the slurry is formed by mixing the anionic surfactants, builders and others. The slurry is spray-dried to obtain a low density detergent stock. The low-density detergent stock and zeolite are introduced to a granulator. Different types of granulators are suitable for this process, such as high-speed mixer densifier (Appel and Swinkels, 1992), pan granulator, concrete mixer or continuous drum mixer (Peter and Thomas, 1991). In this step, zeolite is coated or 'layered' onto the much larger particles of detergent stock (base) powder. The mixing condition should be such as to break up any agglomerate in the zeolites without breaking up the base powder particles. After admixture of zeolite, a liquid binder consisting of nonionic surfactant and sodium silicate are sprayed onto the 'layered' powder. This processing step can be carried out in moderate speed granulator/densifier. This mixer consists of hollow static cylinder having a rotating shaft in the middle (Appel and Swinkels, 1992). In order to obtain the final powder, a cooling step is needed which is carried out in an airlift. Finally, the heat sensitive ingredients such as enzyme, perfume, bleaching agent and bleach activator are introduced to the mixer. Figure 7 illustrates a multifunction processing line for the manufacture of concentrated laundry powder using agitation granulation technology.

Disintegration Granulation

In this method the usage of spray-drying towers are eliminated. This technology has also achieved a significant reduction in energy costs, with lower emissions being discharged into the environment. There is an added advantage to the formulator because the powder ingredients are no longer subjected to the intensive processing conditions usually encountered in spray-drying, particularly with respect to temperature. This means there is less restriction on the use

10

of certain temperature sensitive materials, in particular detergents, such as the fatty alcohol sulphate, ethoxylates, and other useful nonionic derivatives (Masayoshi and Noboru, 1990).

A simplified diagram in Figure 8 shows the steps involved in the production of concentrated powder detergent using this process. The detergent ingredients, surfactants, builders and others are introduced into a kneader to obtain a uniformly kneaded mixture in the form of a sheet. The resultant mixture in the form of a sheet was pelletized in a pelleter to facilitate the disintegration. The disintegrated detergent composition and zeolite are continuously and quantitatively fed to a rolling/mixing granulator. The coated product is discharged and a granular detergent composition having a high bulk density is obtained (Hisako *et al.*, 1993). Figure 9 illustrates a multifunction processing line for the manufacture of concentrated laundry powder using disintegration granulation technology.

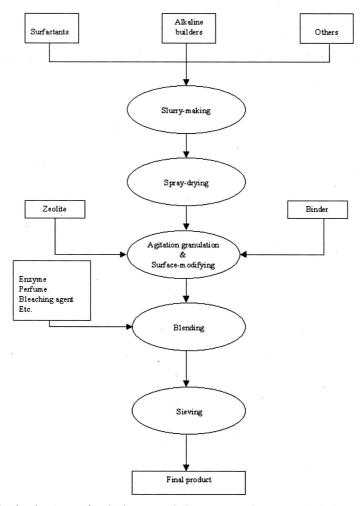


Figure 6. The basic steps of agitation granulation process for concentrated powder detergent

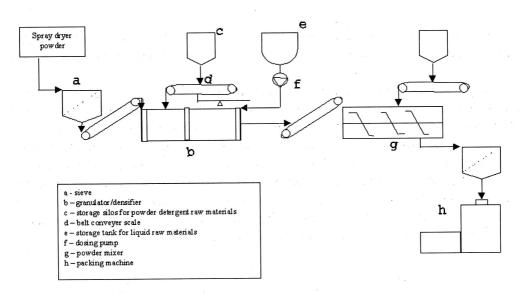
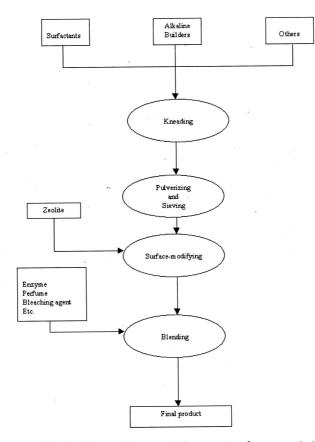
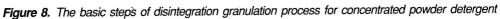


Figure 7. Flow diagram of agitation granulation process for concentrated powder detergent





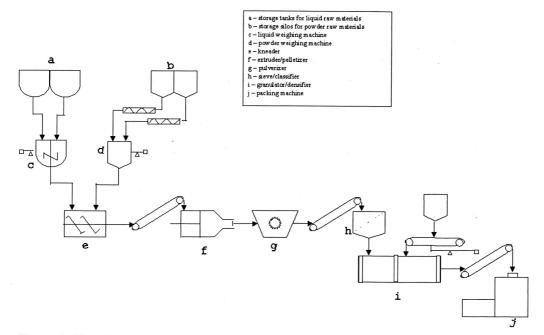


Figure 9. Flow diagram of disintegration granulation process for concentrated powder detergent

REFERENCES

Appel, P.W. and Swinkels, L.J. (1992). U.S. Patent 5, 133, 924.

Coffey, R. and Gudowicz. (1990). Trends in Siliceous Builders. *Chemistry & Industry*, March **19** : pp 169-172.

Croy, C. (1994). INFORM, 5(1) : pp 62-68.

Falbe, J. (1987). Surfactants in Consumer Products, Springer-Verlag Heidelberg, New York.

Herman De Groot, W., Moretti G..F. and Adami I. (1995). The Manufacture of Modern Detergent Powders, Herman De Groot Academic Publisher, The Netherlands.

Hisako, Y. and Hiroshi, N. (1993). U.S. Patent 5,263,650.

Masayoshi, N. and Noboru, H. (1990). U.S. Patent 4,970,017.

Milne, N.J. (1998). Oxygen Bleaching Systems in Domestic Laundry. J. Surfactants and Detergents, 1(2) : pp 253-261.

Peter C.K. and Thomas T. (1991). U.S. Patent 5,030,379.

Ropien, J. (1993). Production of Powder Detergent by Spray-mixing Technique Suitable for Small Scale Industries. *J. Industrial Technology*, **3(1)** : pp 57-64.

Staley, D.S. (1997). U.S. Patent 5,635,467.

Technical Information (1987). Spray-mixed Powder Detergents, BASF.

Thayer, A.M. (1993). Chemical & Engineering News, Jan. 25 : pp 26-47.

Whalley, G. (1995a). SPC, Feb. 1 : pp 41-42.

Whalley, G. (1995b). Manufacturing Chemist, Nov. 1: pp 38-41.

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