results, a judgment is made as to whether any corrective measures are necessary.

Still other samples are exposed to sunlight to simulate storage of a non-opaque plastic bottle near a kitchen window and to investigate sensitivity to artificial light. Fading of color or darkening of product requires that the troublesome components be identified and corrective measures taken.

Whenever any formula corrections are made, the entire series of aging studies should be repeated. It is obvious that unexpected difficulties can easily upset marketing timetables.

Adequacy of Preservation Studies

These are conducted to assess the product's ability to withstand microbiological contamination. Inability to control the growth of key species usually demands incorporation of a suitable preservative.

PRODUCT SAFETY ASSESSMENT

The most important tests run on a finalized product are those that permit assessment of its medical safety. These include animal tests specified by the Federal Hazardous Substances Law, human skin irritation tests, and analyses for presence of contaminants or impurities of concern to government regulatory bodies.

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Formulation of Household Automatic Dishwasher Detergents

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ABSTRACT

The growth of the household automatic dishwasher detergent market and factors affecting future growth is reviewed. Major formulation changes that have occurred during the years are discussed, with emphasis on those contributions which resulted in significant improvement in performance. Present day formulations are classified according to types of ingredients and method of manufacture. Formulation options, types of equipment that can be used and factors which affect product performance are discussed, and performance test methods are described.

INTRODUCTION

Mechanical dishwashers were in use before the turn of the century, but an effective detergent product did not reach the market until the mid-1930s. The early products were soaps or simple mixtures of alkalies which softened water by precipitation. Gross food deposits were flushed away but were replaced by a film of insoluble calcium and magnesium salts.

Several major formulation improvements have occurred during the years to provide the high performance formulations available today (1).

DEVELOPMENT OF HOUSEHOLD FORMULATIONS

The first major improvement in dishwasher detergent occurred in the mid-1930s with the discovery that polyphosphates could be used to complex calcium and magnesium ions and prevent the formation of insoluble films (2,3). A sodium polyphosphate glass (Graham's salt) was used in the first of these products, but eventually was replaced by sodium tripolyphosphate because of its better performance and handling properties.

In addition to its ability to soften hard water, sodium tripolyphosphate is an excellent emulsifier and dispersing agent for soils, and helps hold the soils in suspension so that they can be rinsed freely from the dish surfaces (4).

The next major improvement was the discovery that an available chlorine compound in the formulation could promote free-rinsing and help to eliminate water spotting (5-7). Chlorine is particularly effective in breaking down protein-type soil to soluble amino acids which are more easily removed by the detergent. Without the chlorine, minute particles of residual soil remain on the dishes and glassware and allow droplets of water to remain through the rinse cycle. Upon drying, these droplets leave behind dissolved solids which cause unsightly spots. In addition to elimination of water spotting, the available chlorine compound also provides improved removal of stains and contributes to sanitizing (5).

The first source of available dry chlorine found to give good performance in automatic dishwashing was chlorinated trisodium phosphate. More recently, chlorinated isocyanurates have been used as the source of available chlorine (8). These compounds provide better stability at elevated storage temperatures, less caking tendency and lower formulation cost than chlorinated trisodium phosphate. The first of these products to be used were the sodium and potassium salts of dichloroisocyanuric acid. More recently, sodium dichloroisocyanurate dihydrate was introduced under the trademark "Clearon" dry bleach. This dihydrate has safety advantages in handling and storage and is more stable than the anhydrous sodium salt in agglomerated dishwasher detergents (9,10). Because of the available high chlorine content of the chlorinated isocyanurates (56-64%) compared to that of chlorinated trisodium phosphate (ca. 3.5%) they offer greater formulation flexibility.

The third major improvement in automatic dishwasher detergents was the introduction of low-foam, nonionic surfactants and defoamers. High foam interferes with the mechanical action of the water spray in the dishwasher and results in poor cleaning. The development of low-foam surfactants allowed an increase in efficiency of soil removal and improved free-drainage of the rinse water to help reduce water spotting. In addition to the nonfoaming characteristics of the surfactant it is desirable to inhibit the foaming caused by protein food soils, such as egg and milk solids. Improved surfactant systems were developed in which a small amount of defoamer, such as monostearyl acid phosphate, is added to a compatible low-foam surfactant (11).

Although it is not a new development, another essential component of a high performance automatic dishwasher detergent is sodium silicate. Sodium silicate acts as a corrosion inhibitor for metals and provides protection for fine china glaze and patterns. In addition, sodium silicate provides alkalinity which contributes to overall cleaning performance.

Most of the earlier detergents were dry-blended formulations containing sodium metasilicate. However, the major products on the market now are agglomerated, and sodium silicate solutions are used as the agglomerating agents.

Another ingredient found in many dishwasher detergents is sodium carbonate. This is usually used in conjunction with the chlorinated isocyanurates to provide added alkalinity that would otherwise be supplied by the chlorinated trisodium phosphate.

In the 1960s there was considerable effort to develop inhibitors which would prevent attack of the dishwasher detergent on the overglaze and patterns of fine china. A number of patents were issued covering various aluminum compounds as inhibitors, such as sodium aluminate (12), sodium aluminum phosphate (13), aluminum acetate (14), aluminum chlorhydroxide (15), metallic aluminum (16), and sodium silico-aluminate (17). A particularly effective approach was to add sodium aluminate as a solution sprayed onto the sodium tripolyphosphate (18). This had two benefits: partial hydration of the sodium tripolyphosphate, which is important in eliminating product caking problems, and a reduction in the amount of aluminate needed for inhibiting china attack to only 0.1-0.3%. Another patented inhibitor is boric acid or boric acid anhydride (19). This appears to be the only material currently in use specifically for overglaze protection. Adequate inhibition of attack on overglaze can apparently be achieved by using higher SiO_2/Na_2O ratio polysilicates instead of metasilicate, and this appears to be the approach now used in most formulations.

A number of optional ingredients can be used in automatic dishwasher detergents, including sodium sulfate, sodium chloride, and dyes or perfumes. Sodium chloride has been shown to be a source of corrosive attack on aluminum when used in car wash formulations and it may cause some attack on aluminum ware in automatic dishwashing.

CURRENT HOUSEHOLD FORMULATIONS

The major household automatic dishwasher formulations have shown very little change in composition over the past 15 years except for the trend towards agglomeration with liquid silicates. All states which have legislation banning the use of phosphates in home laundry detergents have exemptions for automatic dishwasher detergents. However, five states (New York, Maine, Michigan, Vermont and Wisconsin) have legislation which limits the phosphorus content of automatic dishwasher detergents to 8.7%, and one state (Minnesota) has a maximal limit of 11.0% phosphorus. These six states and nearby affected regions represent ca. 19% of the total U.S. population. Because of these restrictions, most of the major producers of household automatic dishwasher detergents now supply both higher phosphate and lower phosphate formulations.

Table I shows typical formulations of higher phosphate and lower phosphate products made with either chlorinated trisodium phosphate or a chlorinated isocyanurate as the source of available chlorine. In the lower phosphate formulations, the one made with chlorinated isocyanurate (formulation D) can contain more sodium tripolyphosphate and still meet an 8.7% phosphorus limitation than the one made with chlorinated trisodium phosphate. Over 80% of the total market is made up of formulations similar to those in Table I, made by agglomeration with liquid silicate.

PRODUCT REQUIREMENTS

A properly formulated automatic dishwasher detergent should have the proper bulk density so that the dispenser cups hold the optimal dosage, dispense rapidly and completely from the dispenser cups without caking, have low foaming properties and the ability to inhibit foaming of food soils, remove all forms of food soils, prevent water spotting and filming, not damage or corrode dinnerware, utensils, glasses, or the dishwasher machine, and function properly in a variety of machines and water conditions.

In addition, the product should have adequate shelf-life, i.e., remain free-flowing and not lose available chlorine in storage. This requirement is a function of proper packaging as well as proper formulation.

The property of rapid dispensing without caking in the dispenser cup is very important from a performance standpoint. Most dishwashers have two dispenser cups, one of which remains covered during the first wash and rinse cycles. The detergent in this cup is exposed to the hot humid atmosphere within the machine during the first cycle and becomes damp. If cupcaking, or incomplete washout, occurs during the second wash cycle some of the detergent can remain through the final rinse cycle. A high concentra-

TABLE I

Typical Formulations of Household Automatic Dishwasher Detergents

	Composition (wt %)			
	Higher phosphate		Lower phosphate	
Formulation	A	B	C	D
Sodium tripolyphosphate	45	45	25	34
Sodium silicate (dry basis)	14	14	12	12
Nonionic surfactant	3	2	4	2
Chlorinated TSP	25	–	25	–
Chlorinated Isocyanurate	–	2	–	2
Sodium carbonate	–	25	–	25
Sodium sulfate	–	–	22	13
Hydrate water[a]	13	12	12	12

[a]Does not include water of hydration of chlorinated TSP.

tion of detergent in the final rinse can result in permanent etching of glassware (4).

Cupcaking is generally attributed to insufficient hydration of the sodium tripolyphosphate or too many fine particles in the product. Under the highly humid conditions in the machine, anhydrous sodium tripolyphosphate tends to form hexahydrate crystals which can cement adjacent particles and form a cake in the dispenser cup. This can be avoided in dry-mixed products by using sodium tripolyphosphate hexahydrate instead of the anhydrous salt. In agglomerated products, the sodium tripolyphosphate particles become coated with hexahydrate when sprayed with sodium silicate solution during the agglomeration step. However, even under these conditions, it is claimed that partial prehydration of the sodium tripolyphosphate before agglomeration can be beneficial (20,21).

Excessive fines in the detergent product increase the packing in the dispenser cup and increase the rate of moisture adsorption so that caking is more apt to occur. Dry mixed products should be made from all granular components to avoid this problem. Agglomerated products are generally fairly coarse, and unagglomerated fines usually can be recycled to the process.

A range of compositions covering most automatic dishwasher detergent formulations is given in Table II. As mentioned previously, the essential components are sodium tripolyphosphate, sodium silicate, a source of available chlorine and a low-foaming surfactant.

Sodium tripolyphosphate is generally used at a level of 25-45%, on the anhydrous basis. Performance improves as the tripolyphosphate content is increased. A recent patent (22) indicates that performance drops off drastically if the sodium tripolyphosphate content falls below ca. 20%. Another study has shown that the drop in performance which occurs if the dishwasher is operated at lower-than-recommended temperature, for energy saving, can be partially offset by increasing the sodium tripolyphosphate content (23). A recent study of various organic chelating agents (including sodium citrate and sodium nitrilotriacetate) as potential replacements for sodium tripolyphosphate showed that all of the organic builders tested gave poor performance with respect to spotting and filming of glassware (22). Similar results had been published earlier (24).

For dry-mixed formulations, granular sodium tripolyphosphate hexahydrate usually is used, or else the formulator partially hydrates granular anhydrous sodium tripolyphosphate. In the latter case, it is preferred to use Phase I sodium tripolyphosphate because it hydrates more rapidly than the Phase II variety. The choice of bulk density depends on the density of other components of the formulation as the finished product should have a bulk density in the range of ca. 0.8-1.0 g/ml to provide the proper concentration in the wash water.

For agglomerated products, the sodium tripolyphosphate can be either powdered or granular, depending on the agglomeration process used. In either case, the Phase I variety is preferred for maximal hydrate formation.

Sodium silicate generally is used at a level of ca. 12-15%, on the anhydrous basis. It is important to use enough silicate for protection of metal and dishware surfaces, but high levels should be avoided because they can promote film or haze formation on glassware. If silicate is not completely rinsed from the glassware before drying, it will etch the surface.

For dry-mixed formulations, either sodium metasilicate or one of the spray-dried hydrous silicates, such as "Britesil," can be used. The hydrous silicates are more expensive than metasilicate but, because of their higher

TABLE II

Range of Compositions of Major Household Dishwasher Detergents

	Range (%)
Sodium tripolyphosphate (anhyd basis)	25-45
Sodium silicate (anhyd basis)	12-15
Surfactant	1-5
Available chlorine	0.5-2.5
(either chlorinated trisodium phosphate)	(20-30)
(or chlorinated isocyanurate)	(1-4)
Sodium carbonate	0-35
Fillers[a]	0-35
Optional additives[b]	0-5

[a]Fillers such as sodium sulfate or sodium chloride.

[b]Optional additives such as borates or aluminates for, e.g., china protection, foam suppressers, dyes, perfumes.

SiO_2/Na_2O ratios, they are more effective in protecting metal and dishware surfaces and are less hazardous in case of accidental eye or skin contact.

For agglomerated products, sodium silicate solutions are available in a variety of SiO_2/Na_2O ratios from ca. 1.6 to 3.2. Generally, a ratio of ca. 2.4 is used, but advantages are reported for using blends of higher and lower ratio silicates (22).

Most automatic dishwasher detergents contain either 20-30% of chlorinated trisodium phosphate or 1-4% of a chlorinated isocyanurate, to provide an available chlorine content of 0.5-2.5%. Most products fall in the range of 1-1.5% available chlorine but higher levels are reported to be especially effective in removing starch soils (22), and one of the major brand products contains ca. 2.5% available chlorine.

Most dry-mixed products contain a chlorinated isocyanurate because it represents a lower cost source of available chlorine than chlorinated trisodium phosphate. However, agglomerated dishwasher detergents originally were made only with chlorinated trisodium phosphate because the chlorinated isocyanurates were considered to not have sufficient storage stability. Because the stability of "CDB Clearon" dry bleach in agglomerated dishwasher detergents has been demonstrated (9,10) it has become the source of available chlorine in a number of agglomerated products.

Low-foam nonionic surfactants are used at a level of ca. 1-5%. These products are used as defoaming agents, as well as for their contribution to soil removal and free rinsing. Usually, they are ethylene oxide and propylene oxide condensation products. Some of these surfactants have sufficient defoaming ability toward food soils so that a separate foam-suppressing agent is not needed (22). However, some nonionic surfactants are formulated to contain a small amount of foam suppresser especially for use in automatic dishwasher detergents (11).

Sodium carbonate generally is not used in dishwasher formulations which contain chlorinated trisodium phosphate. However, it is used as a source of alkalinity in all formulations containing chlorinated isocyanurates. Use levels range up to ca. 35%.

Fillers such as sodium sulfate or sodium chloride are used in the lower phosphate content products and those which contain chlorinated isocyanurates. Concentrations of up to 35% sodium sulfate are used. Other additives such as coloring agents and perfumes can be used at appropriate levels.

MANUFACTURING TECHNIQUES

Automatic dishwasher detergents are manufactured either by dry blending or by agglomeration. In dry blending, the individual components are merely mixed together in equipment such as ribbon, paddle or tumble blenders. Sodium tripolyphosphate hexahydrate is very absorptive and can be used to absorb the liquid nonionic surfactant and any other liquid ingredients such as the perfume and dye. If anhydrous sodium tripolyphosphate is used, it should be first partially hydrated by spraying with enough water to convert about half of the material to the hexahydrate to avoid caking. Some grades of sodium carbonate also are fairly absorptive and can be blended with the hydrated sodium tripolyphosphate before spraying on the liquids, if desired. The remaining solid ingredients are then added and the mixture thoroughly blended. The main precaution is to avoid direct contact of the chlorinated isocyanurate with the liquid nonionic surfactant, because this is a major source of chlorine instability (10).

Agglomeration allows the individual components to be formed into granules whose composition is representative of the total detergent mix. The dry components are initially mixed together and then agglomerated by spraying with a liquid silicate solution. The water becomes fixed as hydrate or bound water of the detergent ingredients, thereby producing a dry, granular product. Further operations such as conditioning, drying, screening and grinding may or may not be required.

Agglomeration can be accomplished in any suitable mixing device in which the liquid can be sprayed onto the moving or flowing solids. Processes have been described using a ribbon or paddle mixer (25), a mixing drum (21), a rotating, inclined disc or pan agglomerator (20), and several types of rotating drum agglomerators (10,26). When a stationary mixer is used it is usually necessary to use granular sodium tripolyphosphate, or a mixture of granular and powdered phosphate. When rotating agglomerators are used, granular STPP can be used, but better agglomeration is achieved with powder.

A study of processing variables in the preparation of agglomerated dishwashing compositions containing sodium dichloroisocyanurate dihydrate was reported previously (10). In that study, the solid ingredients (sodium tripolyphosphate, sodium carbonate, and granular "CDB Clearon" dry bleach) were mixed in a horizontal rotary drum agglomerator and the liquid nonionic surfactant and sodium silicate solution were sprayed onto the tumbling bed of solids. Three different mixing sequences were evaluated but all three gave approximately equivalent results. The agglomerated products were dried for 45 min at 50 C to remove ca. 3% of loosely bound moisture and enhance flow properties and chlorine stability. The products showed very little chlorine loss during agglomeration and drying and showed available chlorine stability under accelerated storage conditions equivalent or superior to that of the major commercially available products.

A similar study was made using an O'Brien agglomerator (O'Brien Industries Equipment Co., unpublished data). This is a rotary agglomerator containing an internal cage to provide a falling curtain of solid ingredients on which to spray the liquids. The composition and order of addition were 45% sodium tripolyphosphate, 17% sodium carbonate (Grade 100), 2% sodium dichloroisocyanurate dihydrate (medium granular Clearon dry bleach), 2% nonionic surfactant (Plurafac RA43) and 34% sodium silicate solution. Several sodium silicate solutions varying in SiO_2/Na_2O ratio from 2.0 to 2.58 were tested. After addition of the sodium silicate, the agglomerated products were conditioned by continuing agitation in the agglomerator for 15 min, but no drying step was used.

When moisturized Phase I sodium tripolyphosphate powder (ca. 0.4% hydrate moisture) was used, a dry, free-flowing product having a bulk density of ca. 0.8-1.0 g/ml was obtained even without conditioning. The bulk density varied inversely with the SiO_2/Na_2O ratio of the sodium silicate, from 1.0 g/ml for the 2.0 ratio silicate to 0.8 g/ml for the 2.58 ratio silicate. The use of granular sodium tripolyphosphate resulted in a sticky product with considerable lump formation. The granular feed also resulted in product bulk densities below the desirable range of 0.8-1.0 g/ml. Very little chlorine loss occurred, even though the nonionic surfactant was sprayed directly on the mixture containing the chlorinated isocyanurate. Subsequent storage tests showed that all of the products had good available chlorine stability.

TEST METHODS

Evaluation of Cleaning, Spotting and Filming

Evaluation of cleaning performance of automatic dishwasher detergents is difficult because of the wide variety of food soils and the wide range of conditions of soiling, handling of soiled dishware, and water hardness and temperature encountered in actual use. A cleaning performance test has been published by the Association of Home Appliance Manufacturers as a means of evaluating dishwashing machines (27). This test uses eggs, Wheatena, cereal, reconstituted skim milk, canned spinach, margarine, tomato juice and instant tea as the food soils, with detailed procedures for soil preparation and soiling of dishes, glassware, and silverware. After washing, each article is examined visually and the number of particles of soil remaining is recorded. A formula is used to convert the total soil count data into a Washing Index which can be used to compare the performance of one machine with that of another.

This procedure has been studied by committees of the American Society for Testing and Materials and the Chemical Specialties Manufacturers Association and has been judged to be too complex for use in evaluating detergent products. Most studies of dishwasher detergent performance have been based on evaluation of spotting and filming of glassware.

The ASTM has published a standard method for evaluating performance of automatic dishwasher detergent in terms of build-up of spots and film on glassware (28). Glass tumblers are given multiple washes in the presence of specified food soils and the levels of spotting and filming allowed by the detergents under test are compared visibly. The standard soil is a mixture of margarine and powdered milk, with or without cooked wheat cereal. Significant comparisons are said to require from 5 to 15 cycles. The tumblers are rated for spotting and film on a scale of 1 to 5, with a rating of 1 representing no spots or film and a rating of 5 representing complete covering of spots or heavy film.

The use of this test by the author has shown that, at a water temperature of 60 C, the major commercially available dishwasher detergents give satisfactory low ratings through 15 cycles. A formulation is considered to fail the test if 50% of the glasses become covered with light film or one-half covered with spots in less than 15 cycles.

A problem arises if these methods are used to judge performance under significantly varied washing conditions. A representative of a machine manufacturer pointed out that field experience has shown that washing temperatures significantly below 60 C result in faulty cleaning because of fatty food soils which need high temperature to be melted and emulsified (29). Most of the test soils used in published evaluations do not include these higher melting fats, and conclusions drawn as to acceptable performance at lower wash temperature are invalid. Similar findings are contained in a policy statement issued by another machine manufacturer in July 1978, to the Consumer Product Safety Commission (30).

Dispenser Cup Caking

The tendency of a product to become gummy or to cake in the dishwasher dispenser cup can be measured by adding the recommended amount of product to each of the two dispenser cups and running the normal machine cycle. The machine is opened and the amount of detergent retained by the second cup is estimated after the second wash cycle and after each subsequent rinse cycle (25). For maximal performance, no detergent should remain in the cup after the wash cycle.

A simple screening test for comparing formulations is to measure the washout time under simulated conditions. A dispenser cup from a Whirlpool Model SKP-55-0 dishwasher is clamped with the opening in a horizontal plane and 10 g of the detergent product is placed in the cup. Two ml of distilled water at 60 C is added by drops from a buret, moving the tip to wet the entire surface. After 5 min, the cup is turned 90° to a vertical position and a controlled stream of 60 C distilled water is directed at the center of the cup. The time required to wash all of the material out of the cup is measured. Steady water pressure is obtained by siphoning from a beaker placed in a water bath 3 ft higher than the cup and adjusting the flow with a stopcock inserted in the line. A plastic wash-bottle tip is used as the outlet for the stream of water.

Actual washout times vary somewhat with variables in the physical set-up of the equipment, but generally, a washout time of less than 1 min is evidence that cupcaking will not occur.

Corrosiveness to China Overglaze Patterns

A standard ASTM method has been published for measuring the removal of overglaze patterns from tableware by dishwasher detergents (31). This is an accelerated method utilizing chinaware with an overglaze pattern known to be sensitive to alkaline detergents immersed in a solution of the dishwasher detergent at 95-99 C for three 2-hr periods. The original chinaware pattern specified in this method is no longer available, but a replacement pattern meeting test requirements has been selected (Holiday pattern manufactured by Lenox Inc., Lawrenceville, NJ).

Storage Stability

Available chlorine stability of the dishwasher detergent formulations usually is evaluated by measuring the loss of available chlorine under accelerated storage test conditions. A typical procedure involves the use of 50-g samples of the formulations in 4-oz glass sample bottles in a controlled temperature/humidity oven. Samples are tested under sealed conditions, with the jars covered with nonpermeable plastic caps, and under permeable conditions, with the jars covered with a moisture-permeable polyethylene-paper laminate. Typical storage conditions are 3 to 6 weeks at 37 C, 80% relative humidity for permeable storage and 6 weeks at 32 C and 43 C for sealed storage. Samples are analyzed iodometrically for available chlorine before and after storage to determine the percentage of original available chlorine remaining after storage.

Consumer Acceptance

The ultimate evaluation of overall performance and acceptability of a product is in the hands of the consumer. If a product does not perform up to expectations under the consumer's use conditions, or has some unacceptable property such as disagreeable odor, dustiness, or poor flow properties, it will be rejected. For this reason, it is common practice to subject a new formulation to some form of consumer test. This may be a small panel test in which a group of employees uses the product in the home and compares it with a commercially available product, or it may be a large panel test of volunteer consumers who fill out a questionnaire indicating the favorable and unfavorable characteristics of the product. Regardless of the amount of in-house testing performed on a new product, the consumer will make the final choice in the marketplace which determines the success or failure of the product.

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