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PREMIX MOLDING COMPOUNDS, THE EFFECT OF PROCESSING TECHNIQUE ON PROFITABILITY AND PRODUCT INTEGRITY

BY

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FOR

DEPARTMENT OF CHEMICAL ENGINEERING
NEWARK COLLEGE OF ENGINEERING

BY

FACULTY COMMITTEE

APPROVED: ______________________________

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JUNE, 1968

PREFACE

The study was undertaken basically to give impetus to new process development in the thermosetting molding compound industry. It is hoped that the following text shows that new process routes to replace cumbersome, antiquated techniques are not unattainable, and definitely in line with the never ceasing need to update, improve and modernize. The potential is certainly present, pending the proper engineering approach, and, the benefits could be substantial.

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PREMIX MOLDING COMPOUNDS, THE EFFECT OF PROCESSING TECHNIQUE ON PROFITABILITY AND PRODUCT INTEGRITY

I. ABSTRACT

Premix molding compounds, presently manufactured in a double-armed blade kneader mixer, can now be prepared by a new and unique method utilizing a high speed densifier. Compounds processed in a kneader mixer, by virtue of its overlapping, shear mixing motion, are extremely susceptible to glass fiber degradation and consequent mechanical strength reductions in moldings. Previously, as this was the only equipment available with the capability to admix highly viscous materials, processors would formulate and design to compensate for the resultant property reductions that are encountered. Also typical of conventional kneader mixer processes is an inherent materials' handling problem and mixing cycles proportionate to the viscosities of the resins employed.

It has now been demonstrated on a laboratory scale, utilizing a Beardsley-Piper Speed Densifier, that high intensity mixing can manufacture premix molding compounds more efficiently and economically while exhibiting an improved product in a more handleable form. Mixing is accomplished centrifugally at high peripheral speeds, thus, eliminating the pronounced effect of resin viscosity on cycle time and resulting in a product with minimum fiber degradation and maximum mechanical strength retention.

II. INTRODUCTION

Premix molding compounds, virtually unknown twenty years ago, now account for twenty five percent of all thermoset molding in the United States. This percentage continually increases as new and improved resin-filler-fibrous reinforcement composites are developed to meet the specific requisites of the multitude of new applications that evolve yearly.

Since its inception, premix molding compounds have progressed considerably by major advancements in mold design, molding techniques, and composite formulation experience; however, process innovations in the manufacture of these compounds, to date, have been confined primarily to minor modifications of the antiquated, conventional kneader type mixing equipment which has been utilized throughout the years.

Conventional premix operations employ a kneader mixer with either sigma or spiral blades which have the capability to admix highly viscous materials with glass fibers into a homogeneous, uniform mix. In process compounding, the raw materials are charged throughout the mixing cycle with the last addition comprised of the reinforcing fibers, generally glass. Mixing of the glass fibers is quite critical because they must be mixed long enough to insure thorough dispersion and coverage with the other materials and, as the batch is of a high consistency, mixing must be short enough so that fiber degradation is minimized, consequently, exhibiting moldings with maximum attainable mechanical strength. The kneader mixers accomplish mixing by way of a shear folding-type action which, by its very nature, contributes to fiber degradation. After mixing, the batch, generally in the form of a tacky, nonhandleable mass, is usually extruded into logs which can be cut to a given length corresponding to the charge weight for a subsequent molding application. In extruding these materials, screw type as opposed to ram extruders are generally employed, and to a degree also contribute to fiber degradation. This complete process technique, aside from the normal degradation of product that is encountered, is both cumbersome and expensive from the standpoint of discharging product, handling product, and cleaning the equipment between color changes when varying color is desirable or necessitated by customer preference.

This study proposes the utilization of high intensity mixing equipment to replace the conventional kneader masters. For a comparative investigation versus the conventional technique a Beardsley-Piper Speed Densifier was employed. The densifier accomplishes compounding by centrifugally mixing the various components, and appreciably minimizes the normal fiber degradation that is encountered while discharging a product in a more handleable form (particulate as opposed to bulk). Previously, the Beardsley-Piper Speed Densifier has been utilized to mull sand for castings and, in regard to premix or thermosetting molding compounds, it has been evaluated by some processors on a one or two attempt cursory basis resulting in varied opinions as to the merits of this equipment. Heretofore, no definitive, controlled experimentation has been performed which conclusively demonstrates the overall, potential benefits of one process route versus another.

The following investigation, employing a laboratory Beardsley-Piper Speed Densifier, is intended to demonstrate the potential of and give impetus to the subsequent development on a pilot and production basis of utilizing high intensity equipment for the manufacture of premix molding compounds. A listing including some of the proposed benefits would be as follows:

1. An improved product by virtue of preserving glass fiber integrity.

2. Increased productivity per given mixer capacity i.e. faster cycles.

3. An improved product form, thus, minimizing the conventional materials' handling problem.

4. Potential Versatility - i.e. in addition to processing premix compounds, other thermoset compound types and forms could be manufactured.

III. PRODUCT DESCRIPTION

A premix molding compound is a homogeneous combination of a catalyzed polyester resin, inert fillers, and reinforcing fibers which finds a definite utility in molding, under heat and pressure, many functional, complex and intricate contours. The inherently low cost of premix, as compared to other materials and processes available, continues to give the impetus to the trend to improve premix material properties for wider consideration into applications now carried out by die-casting, sheet metal stamping, and many other conventional systems. Premix techniques allow for a greater variety of fibers and fillers to be utilized in their simplest forms, thus, enabling the rapid molding of varying wall thicknesses, molded-in inserts, holes, slots, grooves and bosses with a resultant cost reduction and minimum waste.

Each component of a premix compound (i.e. resin, filler, fiber etc.) has a definite function, and varying either their proporations or their types, exhibits a line of compounds with a wide spectrum of mechanical, physical and electrical properties for a multitude of applications. In short, a premix molding compound may be defined as being in a B-stage (thermoplastic) with limited stability and, for final processing to the C-stage (thermoset), the material is charged to a heated mold under high pressure. The pressure supplies the force enabling the material to fill the mold cavity while the temperature activates the catalyst which, subsequently, cures the molded part to its final degree of rigidity. By virtue of both its low raw material cost and improved molding techniques which approach the rate of die-casting, premix has made some definite inroads and advancements in replacing parts made from metal. In many instances, through proper design, use of premix has enabled the production of a single molding which previously consisted of an assembly of several metal parts.

Typical applications for premix molding compounds is continually expanding from year to year and becoming more diverse in regard to specific market area. Various markets utilizing premix include the automotive, electrical, electronic and appliance industries. The materials are generally employed for both functional and non functional interior as well as exterior, structural applications. A brief listing of typical premix applications would be as follows:

1) automotive heat-air conditioning housings
2) automotive consoles
3) coil towers
4) distributor caps
5) automotive tail light housing
6) air-filter covers
7) electrical connectors and printed circuit boards
8) encapsulation of electrical components
9) swimming pool filter housings
10) humidifier housings
11) circuit breaker and switch-gear housings
12) timing-chain covers

As previously indicated, a premix molding compound generally contains polyester resin, fillers, reinforcement, catalyst, pigment and an internal mold lubricant. The following will serve as a descriptive guide to the various components such that their relative effect on product properties and processing response can be related to the subject of this study:

1. <u>Resin</u>

The polyester resins utilized in premix molding compounds are formed by the poly-condensation reaction of various dibasic acids (saturated and unsaturated) and poly-hydric alcohols. Generally, maleic anhydride, fumaric acid, phthalic anhydride, isophthalic acid, ethylene, propylene, diethylene and dipropylene glycols in varying proportions are employed. Other specialty type resins such as flame retardant are prepared by incorporating a quantity of halogenated dibasic acids (e.g. tetra chlorophthalic anhydride and chlorendic anhydride). The resultant polyester resin solids are extremely viscous materials and are subsequently dissolved or diluted in a liquid vinyl monomer to lower the viscosity and make the resin more handleable, while also providing a reactive cross linking agent enabling the rapid curing of these systems. Typical vinyl monomers include styrene, vinyl toluene and diallyl phthalate. Styrene and vinyl toluene, due to their volatility, are more suitable for molding compounds which are manufactured and molded within one to two weeks. The high volatility renders poor compound stability. Diallyl phthalate, on the other hand is essentially non-volatile and exhibits resultant compounds with stability upwards to six months. However, the cost of diallyl phthalate is approximately four times that of styrene. Therefore, styrenated polyesters are generally employed by custom molders who captively manufacture their own premix compounds -- typical of the automotive companies -- while compound suppliers utilize diallyl phthalate cut polyesters in order to market materials with adequate stability. Those molders whose compound requirements and types vary considerably are not in a position to effectively and economically manufacture their own compounds, and are restricted to purchasing from suppliers.

In compounding for specific applicational requisites, no one resin is ideal. Part design, service requirements, mechanical and electrical properties must be carefully scrutinized before a particular resin is selected.

In molding, it is the resin which must carry the reinforcement and filler uniformly to all parts of the mold. Generally the higher the resin viscosity the more effectively this is accomplished. However, viscosities that are too high represent both a handling and processing problem. Viscosities for styrene and vinyl toluene cut polyesters generally range from 25-35 poise with 200 poise being the maximum, useful upper-limit. As heat build-up during compounding is directly proportional to mix consistency, resin viscosities greater than 200 poise are prohibitive with volatile monomers. Diallyl phthalate, on the other hand, being non-volatile allows for the use of higher viscosities, and can withstand the accompanying higher processing temperatures without detrimental effects to the product. Resin viscosities with diallyl phthalate monomer of 2500 poise are not uncommon.

2. Fillers

The addition of fillers to polyester resins serve two prime purposes. One is to dry-up the resin and yield a more handleable product from the standpoint of a molding compound; while, the other is to primarily reduce the overall molding compound raw material cost. Fillers range in price from $.01 to $.15 per pound whereas the resins range from $.20 to $.50 per pound. Typical fillers which have found extensive and successful utilization in premix include clay, calcium carbonate, gypsum, talc, mica, and aluminum hydrate. Clays and calcium carbonate find the most widespread usage, basically because of their low cost and satisfactory performance. Clay fillers do exhibit certain advantages such as longer shelf life, and an improved surface appearance on molding. Clay filled systems also exhibit superior resistance to degradation in post-bake cycling, especially in applications requiring painting at elevated temperatures. Clay, however, has a relatively high oil absorption resulting in a rapid increase in mix viscosity, thus, necessitating resin contents on the order of 30-35 percent. Calcium carbonates, on the other hand, possess lower oil absorptions and offer higher loading values without a pronounced effect on mix viscosity. Carbonate filled systems generally contain from 20-28 percent resin, thus, yielding lower raw material costs.

In many applications where flame retardance is a requirement, aluminum hydrate fillers are employed, especially in instances where the economics prohibit the use of specialty flame resistant resins. Aluminum hydrate fillers also impart excellent electrical properties to premix compounds, particularly arc and track resistance.

In practice, premix formulators employ combinations of various type fillers in one optimum formulation per application. The resultant formulation is generally a blend of various fillers with widespread differences in particle size and oil absorption yielding a compound which is an optimum balance between economics, moldability and performance.

3. Reinforcements

When polyester resins are diluted with inert fillers, their mechanical properties are largely reduced, i.e., flexural strength, tensile strength, and elongation decrease while flexural modulus increases resulting in an extremely brittle system. As a means to improving these deficiencies, reinforcing fibers are generally incorporated into the compound. The fibrous reinforcement increases the overall structural characteristics of parts molded from these compounds. Not only does fibrous reinforcement restore a significant percentage of the mechanical properties lost by filler dilution, but it also imparts impact resistance which is a definite requisite in many applications.

Reinforcing fibers for premix compounds include chopped glass, sisal, asbestos, and synthetic fibers such as polyester and nylon. Glass fibers are most extensively employed, primarily because they exhibit the greatest strength per pound of reinforcing material at lowest cost. Optimum performance is obtained with glass fibers one quarter to one half inch in length. Fibers in this length-range exhibit efficient mixing characteristics (rapid wet-out and coverage with fillers, resin, etc.), and excellent molding properties. Theoretically, the mechanical properties increase with longer fiber lengths, however, fibers in excess of one half inch do not disperse easily during the mixing operation and are extremely susceptible to fiber degradation; therefore, the increased benefit of fiber length is never obtained. In addition, long fiber lengths exhibit poor moldability, especially in applications with thin wall sections or delicate inserts -- long fibers impede the compound's flow characteristics.

All glass fibers for polyester premix compounding are specially treated with chrome or silane finishes. The finish enhances the wetting characteristics of the glass, and provides for a chemical linkage between the glass and resin.

The glass contents for premix compounds ranges from 5 to 35 percent. An optimum range in regard to processability, preformability (i.e. compaction) and to flow during molding would be 18 to 22 percent.

As previously mentioned, asbestos fibers are reinforcing, however, their effect on mechanical properties is only minor. The real importance of asbestos is utilizing it in part with glass and the remaining resin-filler components. It has an inherent ability to hold the filled, composite system in homogeneity during flow in molding. This results in part uniformity and a minimum of defects and cracking that occur when the various components of the compound separate under high heat and pressure during flow.

4. Catalyst

The majority of premix molding compounds are catalyzed with benzoyl peroxide. This catalyst offers low cost and excellent performance for most applications. Other expensive catalyst types, such as tertiary butyl perbenzoate and peroctoate, are used for specific, applicational purposes to provide longer flow times and extended stability. The peroxide catalysts employed are activated only by the high temperatures (280-350°F) encountered in molding, therefore, ambient or moderately high mold shop temperatures (90-100°F) are not detrimental to compound stability.

5. Pigments

Pigments are employed in premix compounds to impart a variety of colors to moldings depending upon the end-user's preference. Black iron oxide and titanium dioxide are most widely used. Both dry powdered pigments as well as pigment pastes in a variety of colors are utilized. One important precaution which must be exercised when selecting a pigment is to insure that the pigment in question remains inert when combined with the resinous component. Many pigments contain certain metallic salts which, when in contact with the resin, have a deleterious effect on compound stability and curing characteristics. For example, carbon black inhibits the cure of polyester resin; resultant moldings cannot cure to their final degree of rigidity and maximum property levels.

6. Lubricants

Lubricants such as zinc stearate, calcium stearate, aluminum palmitate, and magnesium stearate at a 0.5 to 1.5 percent level are incorporated into premix molding compounds to provide release from the mold cavity. By virtue of their lower specific gravities, the release agents rise under heat and pressure forming a protective film on the molded part which enhances mold release.

IV. EQUIPMENT & PROCESS DESCRIPTIONS

As premix and related molding compounds are extremely viscous materials, the selection of suitable equipment to efficiently manufacture them has always been quite limited. The following outlines both the conventional equipment and procedures currently employed for premix manufacture, and also outlines the proposed new method of manufacture:

1. Conventional Processing

Conventional mixing equipment utilized to manufacture premix compounds is generally referred to as a double-arm kneader mixer. It consists of a rectangular - trough shaped shell equipped with two sigma or spiral shaped blades which rotate approximately 20-35 RPM. The blade clearance, or the distance between blade edge and shell (mixer base) is generally maintained between one quarter to three eighths of an inch. Both blade clearance and blade RPM are critical, and are directly related to product integrity and process efficiency. If blade clearance is too small (less than ¼") and/or RPM is too high, resultant mixing cycles are too rapid, excessive heat build-up is encountered, and glass fiber degradation becomes more pronounced. On the other hand, if blade clearance is too high and/or RPM is too low, undesirable extended mixing cycles result, and therefore, productivity is largely reduced.

A typical procedure for compounding a premix molding compound would consist of the following:

	Process Description	Time (Mins.) For Resins With 15-500 Poise Viscosity	Time (Mins.) For Resins With Viscosities Greater Than 500 Poise
1.	Add all dry filler (i.e. clay, carbonate, lubricant, etc.), and blend thoroughly.	2-5	2-5
2.	Thoroughly pre-mix the resin, catalyst and pigments, and then add to blended fillers. Mix thoroughly until a uniform putty is achieved. Note: Pre-mixing of these three components is applicable for resin viscosities of 10-500 poise. With resins of 500 poise viscosity or greater, all components must be added separately to the Kneader Mixer, and resultant cycles are largely increased.	5-8	8-20
3.	Add fiber glass or other fiberous reinforcement carefully to prevent agglomeration and resultant poor wetting and dispersion.	2-4	4-10

	Process Description	Time (Mins.) For Resins With 15-500 Poise Viscosity	Time (Mins.) For Resins With Viscosities Greater Than 500 Poise
4.	Mix glass fiber thoroughly i.e., long enough to insure complete coverage but short enough to minimize fiber degradation. This time is critical and is directly related to moldability and mechanical property performance. The optimum time varies depending upon both compound type and formulation. It is established empirically by periodic sampling, molding and mechanical property evaluations during this time interval.	5-8	8-12
	Typical Total Cycle Time	14-25	22-47

Since the materials are extremely viscous, substantial heat build-up is encountered. Batch temperatures should not be allowed to exceed 125°F for compounds with styrenated or vinyl toluene cut polyesters, while diallyl phthalate types have a 160°F maximum temperature limitation. In the case of compounds containing volatile monomers, temperatures above 125°F cause excessive evaporation and a resultant stiffness increase which affects the compound's molding plasticity. With the non-volatile diallyl phthalate types, the main factor is not to partially initiate the catalyst to prevent subsequent stability problems. A good processing practice to minimize as many variables as possible is to jacket the mixer, and continually circulate tempered water (90-110°F) through the jacket. This facilitates initial compounding (prior to glass addition) and prevents the mixer shell from becoming "too hot" during constant production - i.e., it eliminates erratic production cycles and batch to batch product variations.

The discharged product from the kneader mixers is in the form of a tough, tacky, unhandleable mass which presents problems from the standpoint of weighing charges at the press during molding. Therefore, to facilitate handling many captive compounder -- molders, and all compound suppliers extrude the premix into slugs of a given length. The uniform slug length corresponds to a charge weight for a given molding application. Screw type as opposed to ram type extruders are generally employed for premix system. The screw compression ratio is 1:1 and, to a degree, also contributes to glass fiber degradation.

The discharging and cleaning of the kneader mixer is both costly and cumbersome. Two thirds to three quarters of the batch can be discharged by rotating the blades with the mixer tilted 90° while remaining material must literally be shoveled out by an operator. Cleaning is extremely time consuming, especially between color changes -- an operator must clean the shell and blades thoroughly to prevent color contamination.

2. Proposed High Intensity Processing

This study proposes the utilization of "high intensity" mixing as a replacement for kneader mixing, in particular a Beardsley-Piper Speed Densifier. This unit commonly referred to as a speed mullor is primarily used to mull sand for castings. It is essentially a cylindrical shaped vessel with a cross-head two thirds up from the base of the vessel pivoted on a central shaft. Attached to the cross-head are two circular rollers which rotate with the cross-head and also about their own axis. The roller clearance between the walls is adjustable. Also attached to the central shaft extending outward along the base of the vessel are two scraper blades. The angle between the base of the mixer and the scraper blades can be varied. Mixing is achieved by the centrifugal motion of the charged formulation between the rollers and scraper blades rotating at high RPM (150-300). The scraper blades force the raw materials at speed between rollers where they are dispersed under high shear. Another unique feature of this equipment is the method of discharging product by way of an air operated door at the base of the mixer.

Successful compounding of premix materials cannot be realized by utilizing the equipment as it is supplied by the Beardsley-Piper Division, Pettibone Mullikan Corporation, Chicago, Illinois. The peripheral speed of the cross-head tip (i.e. RPM per mixer diameter) is too high, resulting in rapid and excessive heat build-up (190-210°F) which consequently causes gelation of the product. It was found that by reducing the mixer RPM by 30% (the diameter of the motor drive pulley was reduced) yields an efficient and unique processing technique for premix molding compounds -- with an acceptable discharge temperature (125-160°F). In practice, it would be advisable to equip this unit with a variable speed drive to impart a degree of versatility to the process. This is especially important when considering the multitude of resin types and formulations which could be processable. This will be discussed further in the conclusion.

A typical procedure for the Densifier would be as follows:

	Process Description	Time (Minutes)
1.	Charge fillers, pigments, lubricant, resin and catalyst and mix thoroughly	2-7
2.	Charge glass while mixer is running and thoroughly disperse	3-5
	Total Cycle Time	5-12

The ranges in cycle times are not as dependent upon resin content, viscosity, and glass content as is typical of kneader mixing.

A compound discharged from the Densifier is vastly superior to the same product discharged from a kneader mixer. Aside from a definite reduction in cycle time, the Densifier exhibits:

1) high mechanical strength properties as a result of the centrifugal mixing which preserves the glass fiber integrity and minimizes degradation.

2) a more handleable product form -- particulate as opposed to a bulk mass -- which discharges rapidly, and can be weighed or fed to an extruder more readily.

3) the potential to be efficiently cleaned by pumping a methylene chloride - cellulose solution to the unit and mixing with high RPM.

V. EXPERIMENTAL PROCEDURES

To prove or disprove the claim of improved mechanical properties by preserving fiber integrity as well as increased productivity for the Densifier over the Kneader mixer, controlled tests were set up to comparatively check these characteristics with a series of formulations.

1. Equipment

Laboratory equipment was employed for experimenting with these mixers. A 2.5 gallon working capacity (15# premix) kneader mixer (J. H. Day Company, Cincinnati, Ohio) was used for conventional processing. A 15 pound laboratory Beardsley-Piper Laboratory Densifier, Model L-50, was used to demonstrate the feasibility of high intensity mixing.

Unfortunately, since the internal mixing construction of the Densifier is complex, laboratory models are not an exact geometric scale - down of production models. The laboratory model has only one roller and one scraper blade, however, the mixing motion or technique is similar and serves to demonstrate the merits of centrifugal compounding.

Since the facilities of Allied Chemical Corporation were utilized for this study, the availability of a semi-production Beardsley-Piper Densifier, Model-400, enabled one, 100 pound trial run to substantiate laboratory results, as well as give supporting data and possible insights into pilot or production scale up.

2. Method

In planning the experiments to compare the product integrity of the identical formulations discharged from these mixers, it was decided that included in the comparison should be property values that could be obtained if no degradation was encountered. Therefore, for most formulations tested, solvent compound processing was included. For these tests the formulation was diluted in methylene chloride and mixed with essentially no degradation in a Hobart Mixer. The resultant product was dried on trays to remove all solvent prior to molding for property evaluations. This, in effect, gives a compound with no degradation and maximum utilization of fiber integrity. Property values from solvent preparations are, in essence, "target values" for the dry mixing systems, and also a relative means to rating the performance of the two mixers in question.

The following comparative tests were performed (in all cases mixing technique is as described in the Process Description):

a) The effect of increasing glass content (¼" fibers) on impact strength, flexural strength, and flexural modulus for kneader, densifier, and solvent processing.

b) The effect of increasing glass fiber length (1/8", ¼", and ½") on impact strength, flexural strength and flexural modulus for a specific formulation in kneader, densifier, and solvent processing.

c) The effect of resin viscosity on mixing cycle time with a specific formulation for kneader and densifier processing.

The mixing method and cycle times per mixer type (i.e., kneader and densifier) for experimenting and testing of compounds processed for the "Glass Content" and "Glass Fiber Length" study were held constant.

For evaluating the effect of glass content on the product properties of kneader versus densifier products, one, typical set of formulation components were utilized. The resin, catalyst and lubricant percentages were held constant while the glass to filler ratio was varied to show the effect of glass content from 0 to 20 percent.

In comparing the effect of glass fiber length on the properties of compounds produced by way of the two process routes in question, the twenty percent formulation from the glass content study was selected for experimenting. Three compounds were produced by each processing scheme from this basic formulation with three, conventionally used, glass lengths - 1/8", ¼", and ½".

By trial and error techniques, prior to initiating the controlled tests, a suitable and efficient mixing cycle was established for both processes. It was felt that the best approach for this type of feasibility study of assessing the relative merits of the densifier and projecting future benefits would be to maintain a constant cycle time. The constant cycle time, however, only yields results which allow the relative performance of the two mixers to be established, and cannot be considered optimum performance with a given formulation for either unit. In fact, with the constant cycle time control, low glass fiber content formulations will be mixed longer than necessary to attain wet-out and, therefore, exhibit some unavoidable fiber degradation.

The procedure employed in this study for kneader processing was:

	Process Description	Time (Minutes)
1.	Charge fillers and lubricant, and thoroughly blend.	4
2.	Charge pre-catalyzed resin. Allow to mix to a homogeneous paste.	7
3.	Charge glass fibers over a one minute interval and mix until complete fiber coverage is attained.	8
	Total Cycle Time	19

Densifier compounds were processed in the following manner:

	Process Description	Time (Minutes)
1.	Charge fillers, lubricant, catalyst and resin. Allow to mix until homogeneous.	4
2.	Charge fiber glass and disperse until thoroughly wet-out.	4
	Total Cycle Time	8

As the time interval between compounding and molding was not always consistent, (i.e. one day to two weeks maximum) a diallyl phthalate cut polyester resin was selected for this program. The prime reason for this selection was that diallyl phthalate, polyester premix compounds exhibit adequate storage stability and a minimal change in stiffness or molding plasticity on aging. With the erratic time lapse between mixing and molding this was the only means to minimizing any molding variables which might have affected the results of these evaluations. If, on the otherhand, a polyester resin containing a volatile monomer had been employed, a significant change in compound stiffness (as a result of monomer evaporation) would have occurred; subsequently affecting the compound's flow (molding) characteristics and resultant mechanical properties.

All products discharged from the mixers were stored at ambient temperature (72-80°F) until molded.

All test specimens were molded in compression molding presses for the subsequent mechanical property evaluations by American Society for Testing Materials (ASTM) methods. Each specimen (flexural strength and modulus, and izod impact) was molded at 1500 pounds per square inch and 300°F for two minutes.

To demonstrate the reduced effect of viscosity on mixing cycle time that is inherent in high intensity equipment, three batches were processed in both the conventional kneader mixer and the densifier with one, specific, compound formulation, incorporating three resins of varied viscosities for comparison. The results of this comparison were based qualitatively on visual inspection of the batch. The end-point, in regard to cycle time, was taken at one minute after which all the dry, uncovered glass disappeared into the mix. Actually, in optimization a series of batches of the same formulation would be manufactured and discharged at progressive, periodic intervals. Mechanical property and molding tests would ascertain the optimum cycle time.

It should be noted that laboratory Densifier models allow for visual batch inspection during process; production models do not.

3. Formulations

a) Formulations for the effect of glass content (¼") on the mechanical properties for solvent, kneader, and densifier processing:

Components	WEIGHT PERCENT				
	A	B	C	D	E
"PLASKON" Polyester Resin 9121 (400 poise, DAP monomer)	23	23	23	23	23
Benzoyl Peroxide - Catalyst (50% in tricresyl phosphate)	1	1	1	1	1
Zinc Stearate-Lubricant	1	1	1	1	1
Drikalite Calcium Carbonate - Filler	67	62	57	52	47
Asbestos, 7T5 - Reinforcement	8	8	8	8	8
Owens Corning Fiberglas - Reinforcement (¼" chopped strand, 841)	0	5	10	15	20
	100	100	100	100	100

"PLASKON" -- Tradename Allied Chemical Corporation

b) Formulations for the effect of glass fiber length on mechanical properties for solvent, kneader, and densifier processing:

Components		WEIGHT PERCENT	
	F	G	H
"PLASKON" Polyester Resin 9121	23	23	23
Benzoyl Peroxide	1	1	1
Zinc Stearate	1	1	1
Calcium Carbonate	52	52	52
Asbestos	8	8	8
Glass 1/8"	15	--	--
¼"	--	15	--
½"	--	--	15
	100	100	100

c) Formulations for the effect of resin viscosity on cycle time for kneader and densifier processing:

Components		WEIGHT PERCENT	
	I	J	K
"PLASKON" PE-750, 1000 poise	25	--	--
"PLASKON" 9121, 400 poise	--	25	--
"PLASKON" PE-336, 25 poise	--	--	25
Benzoyl Peroxide	1	1	1
Calcium Carbonate	45	45	45
Asbestos	8	8	8
Glass, ¼"	20	20	20
	100	100	100

VI. RESULTS

The results of the controlled tests establish that the high intensity based mixing of the Beardsley-Piper Densifier is a vastly superior method for manufacturing premix molding compounds. Mechanical property evaluations indicate that the glass fibers are preserved and, while preserving their integrity, they impart higher property levels. It appeared from the mechanical property results that the range of values obtained from a specific formulation were not only significantly higher but also not as broad as those observed from solvent and kneader processing. However, this observation is based only on a sample of ten tests per property per formulation and, to conclusively substantiate this, more testing with a statistical analysis would have to be performed.

Utilizing the solvent processing technique to represent ultimate, target, zero-degradation property values did not prove to be as successful as was intended. The values obtained were neither consistently nor substantially greater than densifier production and, at times, even kneader production. This could be attributed to one of the following:

1) solvent entrainment - causing polymer softening or cure inhibition and resultant property reductions.

2) poor moldability - maximum preservation of fiber integrity renders a stiffer molding compound for a given control formulation which, subsequently, does not densify properly when molded, and yields lower property values. NOTE: This was evidenced by checking the flow characteristics of each compound manufactured. Utilizing a spiral flow mold, which measures relative flow lengths of various compounds at a given temperature (300°F) and transfer pressure (1500 psi), proved that the

order of decreasing stiffness (resistance to flow in molding) would be solvent, densifier, and kneader compounds.

While impact and flexural strength evaluations exhibit definite product improvements at all fiber glass contents and fiber lengths, flexural modulus (rigidity) results do not indicate any measurable differences.

The controlled tests to determine the effect of viscosity on cycle time proved that densifier production cut cycles by more than 60%. This represents two to three times the output of kneader production at equivalent mixer capacity for continuous production. Hence, on a "grass roots basis", since kneader and densifier units are comparably priced, the economics by way of production capability more than favors densifier investment for plant improvements or new plant installation.

As previously mentioned, the availability of a semi-production Densifier unit enabled a 100 pound trial run. This run exhibited a compound with properties within those ranges obtained for laboratory densifier preparations. Compressive and tensile strength properties were also obtained on this batch, and the values compared more than favorably with those generally obtained with kneader processing of a similar formulation. Besides allowing for the observation of densifier processing on a production size model, this particular trial discharged an improved product from even that of the laboratory densifier -- in regard to product form. As was discussed in the equipment description section, production densifiers differ slightly from laboratory models in construction. Production models have two rollers and two scraper blades; laboratory models have one of each. Products discharged from the laboratory densifier remain somewhat unhandleable --- similar to kneader products. Production models, however, it appears discharge the product in a particulate as opposed to bulk form. This, in effect, yields a product which can be handled more economically and rapidly, i.e. when discharging from the mixer, feeding an extruder, or weighing unextruded material prior to molding. Therefore, potential improved handleability represents a real, definite and substantial savings in processing costs.

The following pages will summarize categorically the results of all tests that were performed in this comparative study.

TABLE I

THE EFFECT OF GLASS CONTENT ON IMPACT STRENGTH FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 24)	IMPACT STRENGTH, FT.-LBS. IN. NOTCH ASTM D 256-56 FROM VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
A, no glass	0.40	0.40	0.40
B, 5% glass (¼")	1.80	1.50	1.20
C, 10% glass (¼")	3.20	3.10	2.60
D, 15% glass (¼")	3.60	3.90	2.90
E, 20% glass (¼")	4.30	4.00	3.20

NOTE: Reported values are an average of ten tests rounded off to the nearest tenth of a foot-pound.

FIGURE I

THE EFFECT OF GLASS CONTENT ON IMPACT STRENGTH FOR VARIOUS PROCESS TECHNIQUES

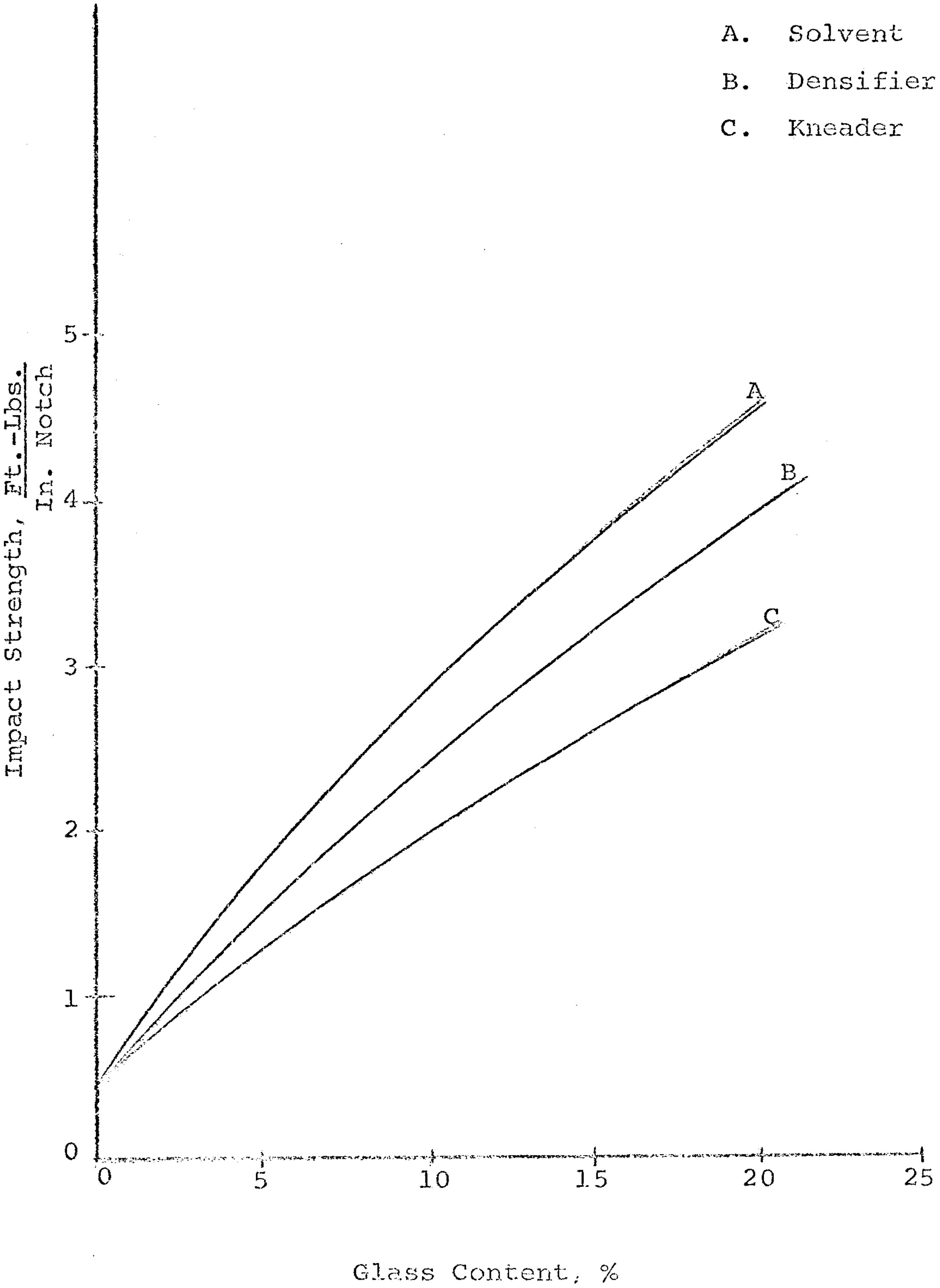

TABLE II

THE EFFECT OF GLASS CONTENT ON FLEXURAL STRENGTH FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 24)	FLEXURAL STRENGTH, PSI ASTM D 790-58T FROM VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
A, no glass	7300	7500	7600
B, 5% glass (¼")	9000	9200	8500
C, 10% glass (¼")	10,000	12,800	10,900
D, 15% glass (¼")	12,300	14,500	12,500
E, 20% glass (¼")	15,000	14,400	13,700

NOTE: Reported values are an average of ten tests rounded off to nearest hundred psi.

FIGURE II

THE EFFECT OF GLASS CONTENT ON FLEXURAL STRENGTH FOR VARIOUS PROCESS TECHNIQUES

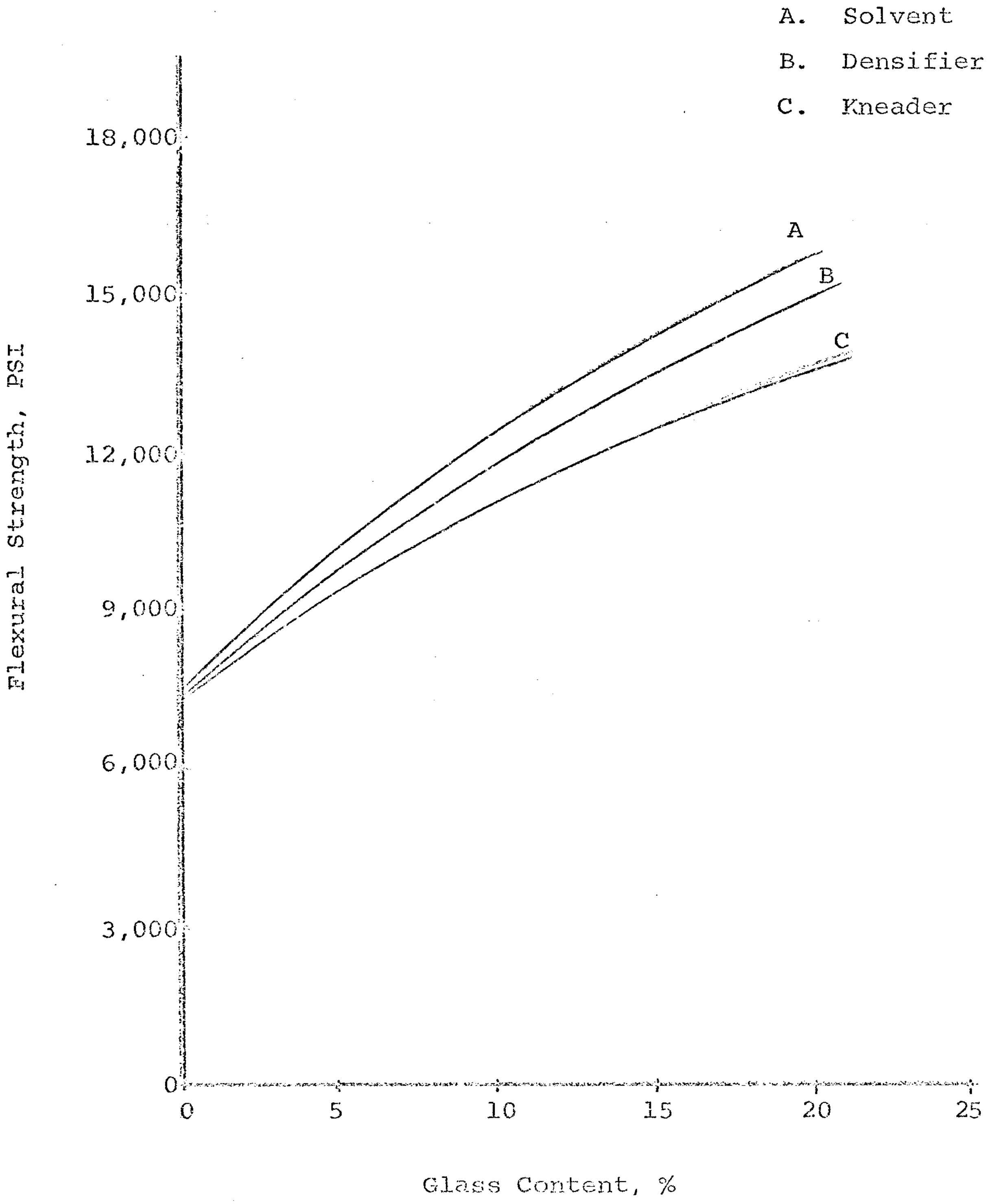

TABLE III

THE EFFECT OF GLASS CONTENT ON FLEXURAL MODULUS FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 24)	FLEXURAL MODULUS, PSI x 10^{-6} ASTM D 790-58T FROM VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
A, no glass	1.30	1.30	1.30
B, 5% glass (¼")	1.70	1.75	1.50
C, 10% glass (¼")	1.85	1.65	1.70
D, 15% glass (¼")	2.00	2.10	1.95
E, 20% glass (¼")	2.00	2.00	2.00

NOTE: Reported values are an average of ten tests rounded out to the nearest fifty-thousand psi.

TABLE IV

THE EFFECT OF GLASS FIBER LENGTH ON IMPACT STRENGTH FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 25)	IMPACT STRENGTH, FT.-LBS. IN. NOTCH ASTM D 256-56 FOR VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
F, 1/8" fibers	2.9	2.6	1.7
G, ¼" fibers	3.6	3.9	2.9
H, ½" fibers	6.3	5.4	3.5

NOTE: Reported values are an average of ten tests rounded off to the nearest tenth.

FIGURE III

THE EFFECT OF GLASS FIBER LENGTH ON IMPACT STRENGTH FOR VARIOUS PROCESS TECHNIQUES

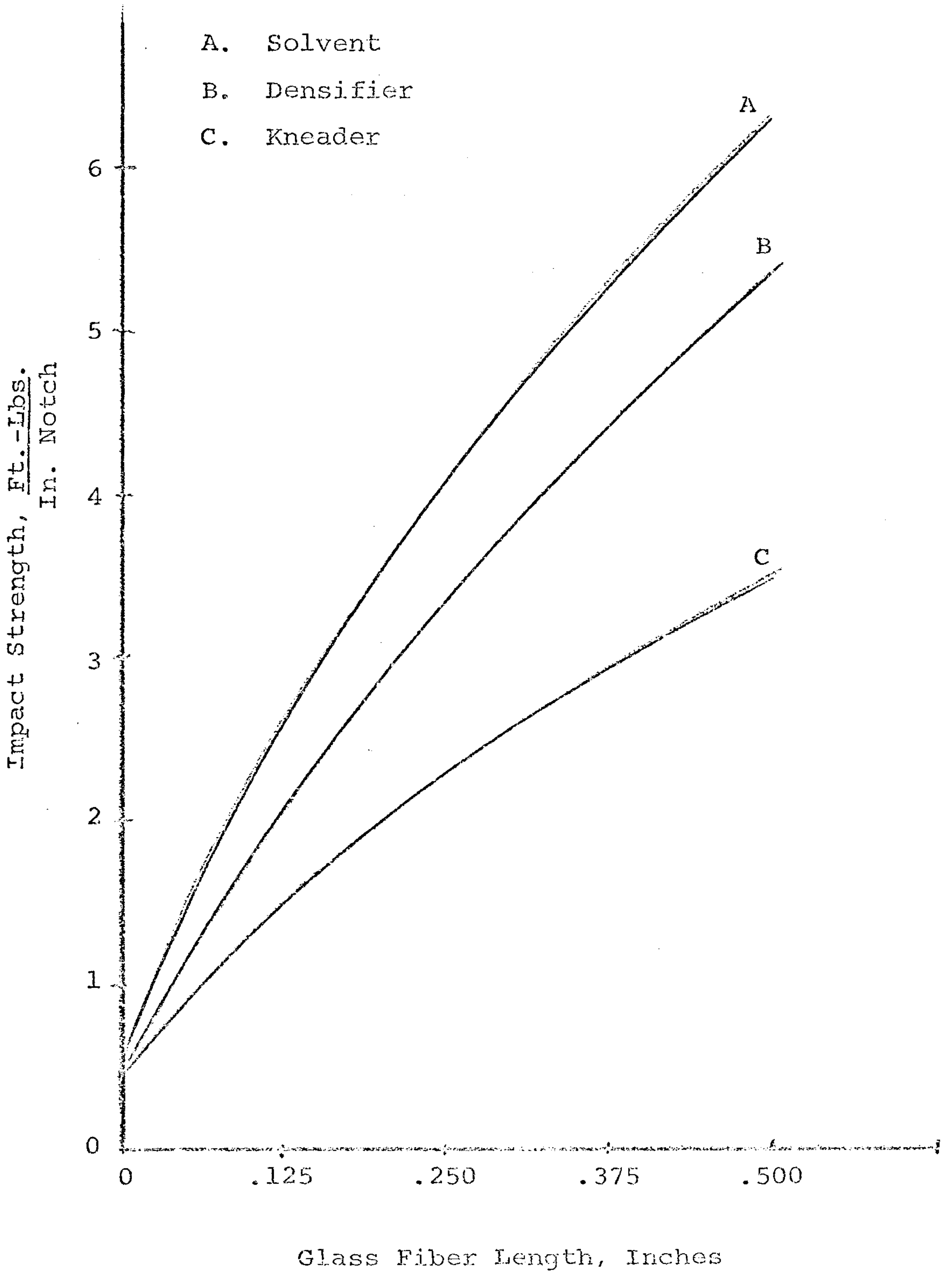

TABLE V

THE EFFECT OF GLASS FIBER LENGTH ON FLEXURAL STRENGTH FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 25)	FLEXURAL STRENGTH, PSI ASTM D 790 58T FOR VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
F, 1/8" fibers	13,200	12,700	10,600
G, ¼" fibers	12,300	14,500	12,500
H, ½" fibers	14,200	15,800	14,100

NOTE: Reported values are an average of ten tests rounded off to the nearest hundred psi.

FIGURE IV

THE EFFECT OF GLASS FIBER LENGTH ON FLEXURAL STRENGTH FOR VARIOUS PROCESS TECHNIQUES

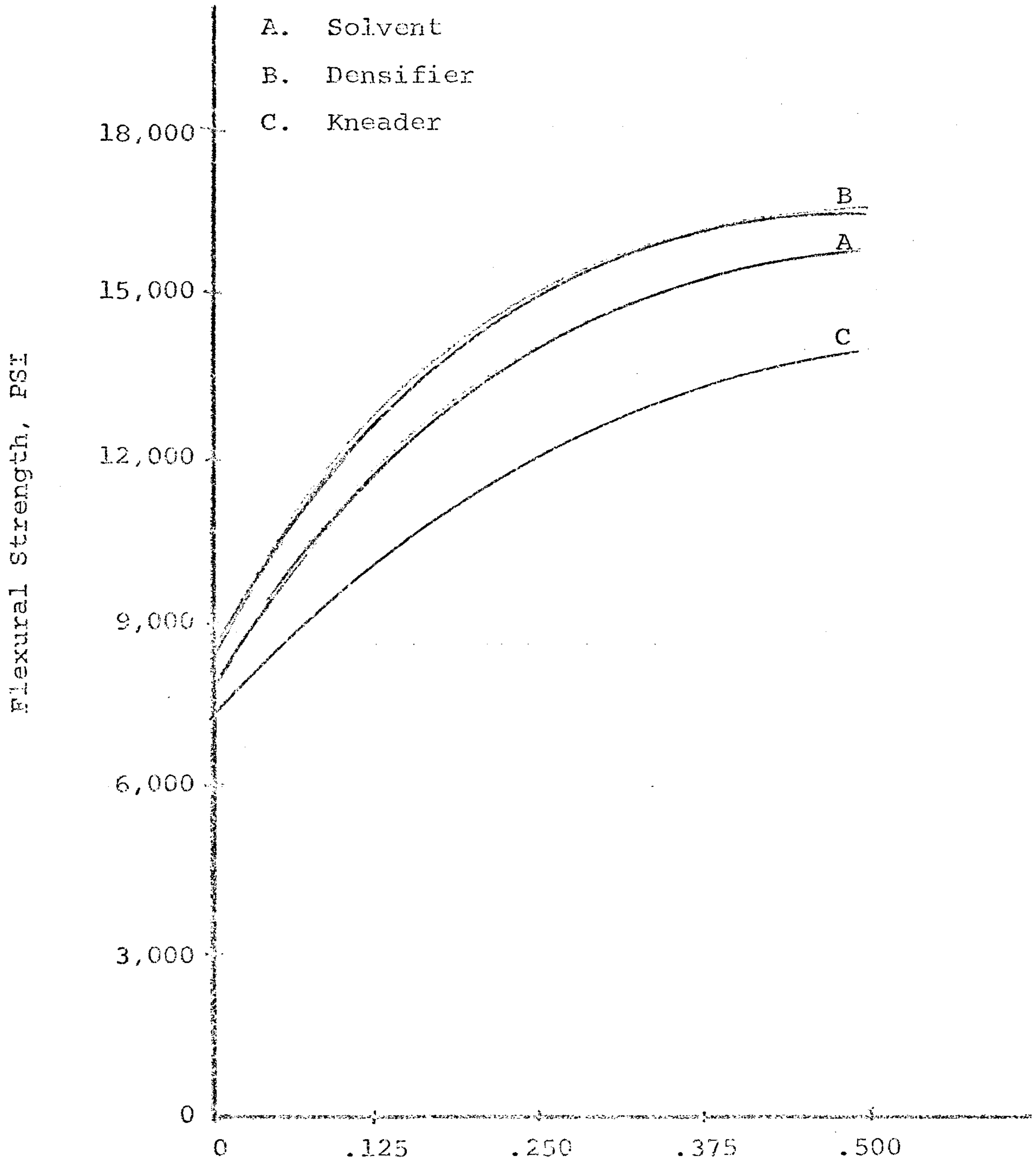

TABLE VI

THE EFFECT OF GLASS FIBER LENGTH ON FLEXURAL MODULUS FOR VARIOUS PROCESS TECHNIQUES

FORMULATION (Reference page 25)	FLEXURAL MODULUS, PSI x 10^{-6} ASTM D 79-58T FOR VARIOUS PROCESSING		
	SOLVENT	DENSIFIER	KNEADER
F, 1/8" fiber	1.75	1.55	1.50
G, ¼" fiber	2.00	2.10	1.95
H, ½" fiber	1.90	2.00	2.00

NOTE: Reported values are an average of ten tests rounded out to the nearest fifty-thousand psi.

TABLE VII

THE EFFECT OF RESIN VISCOSITY ON MIXING CYCLE TIME FOR DENSIFIER AND KNEADER PROCESSING

FORMULATION (Reference page 25)	RESIN VISCOSITY POISE	CYCLE TIME (MINUTES) FOR BOTH PROCESSES	
		DENSIFIER	KNEADER
I	1000	12	45
J	400	8	25
K	25	5	15

NOTE: As previously pointed out, this is basically a qualitative test based on visual batch inspection.

FIGURE V

THE EFFECT OF RESIN VISCOSITY ON MIXING CYCLE TIME FOR DENSIFIER AND KNEADER PROCESSING

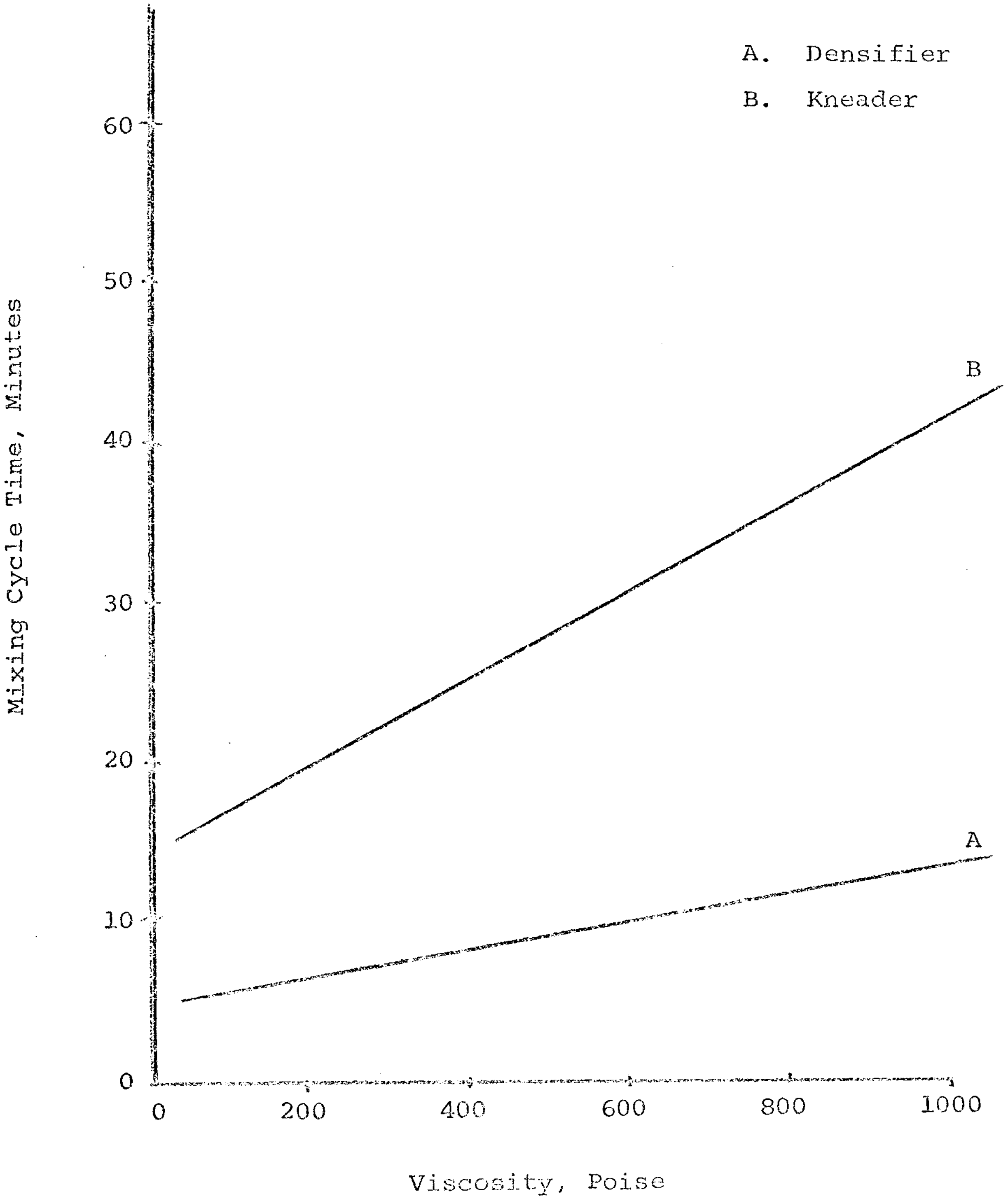

TABLE VIII

TYPICAL MECHANICAL PROPERTIES OF FORMULATION C (PAGE 24) PRODUCED IN A SEMI-PRODUCTION DENSIFIER UNIT, MODEL - 400 (100#)

Izod Impact Strength, Ft.-Lbs. / In. Notch	3.2	(2.5)*
Flexural Strength, psi	13,500	(12,000)*
Flexural Modulus, psi	2.1×10^6	(2.1×10^6)*
Tensile Strength, psi	8,800	(8000)*
Compression Strength	29,550	(30,000)*

* Typical values for Kneader production with this formulation.

VII. CONCLUSIONS & RECOMMENDATIONS

Beardsley-Piper processing to manufacture premix molding compounds definitely exhibits the potential to be an innovative, economical technique for the thermoset industry. Together with significantly reduced cycle times, ease of product handling by virtue of a more desirable product form, and the improved product integrity yields a process with favorable economics and processing versatility, i.e., the capability to process both rapidly and efficiently many diverse formulations which are ever prevalent in the dynamic thermoset industry. From a manufacturing cost standpoint the following benefits can be realized:

1) greater production capability at equivalent mixer capacity and comparable dollar investment -- two to three times the output at equal mixer ratings.
2) due to the high horsepower, high shear mixing, the possibility exists to increase batch sizes at an equivalent capacity rating -- thereby making the economics more favorable.
3) an improved product form yielding reduced and more efficient materials' handling, therefore, reducing labor costs and overall manufacturing costs.
4) production versatility -- the potential exists to produce other thermoset compounds by applying a combined process --- product approach to development, i.e. balancing the formulation variables and process variables to yield compounds

with proper moldability and processability.

It is recommended that any further development with the Beardsley-Piper be performed in the fifty pound unit (Model 20) equipped with a variable - speed drive for processing versatility and latitude. This is necessitated when considering that Model 20 is geometrically similar to production units, therefore enabling a more realistic tabulation and observation of the processing parameters for potential scale-up.

Recommendations for further process development with the Beardsley-Piper Speed Densifier, Model 20, would include the following:

1) determine the optimum RPM per formulation type (i.e. resin viscosity, resin content, glass fiber length and content) in relation to minimum cycle time, maximum product property values and acceptable batch temperatures (i.e. effect of temperature on product stability).
2) determine optimum batch size per formulation type in relation to productivity, properties, and acceptable power requirements for mixer size and construction.
3) explore the possibility of automating the complete premix process --- the improved product form allows rapid discharging of product directly to (by way of conveyors, screw feeders or modified hoppers) the extruding operations.
4) explore the feasibility of processing other thermoset compounds with this mixer or other high intensity equipment that is available in

the field (e.g. Phenolic, Amino, Epoxy and Diallyl Phthalate molding compounds).

The thermoset industry is comprised of a multitude of complex product types and formulations within a given type. Therefore, it is as essentially impossible to divorce product development from process development. Since new process development does in many cases necessitate slight formulation changes, it is imperative that thermoset process engineers have an intimate understanding of the various formulation components and their effect on the compound's performance in end-use applications. Adherence to this philosophy, hopefully, will render many a new and economical process to replace the conventional, antiquated techniques that presently exist, impeding, to a degree, the growth potential of this industry.

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