COMPANY CONFIDENTIAL

S P E C I-A L T Y P R O D U C T S

1 9 7 2 R E S E A R C H A C C O M P L I S H M E N T S

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Edited by: W. R. Richard

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H I G H L I G H T S

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SPECIALTY PRODUCTS RESEARCH

ACCOMPLISHMENTS IN 1972

Key Research Highlights

I. Fluids

* Reformulated Pydrauls

* New NC, N_2C phosphate ester process, AR project and manufacture

* Santosol 100 - product definition, process, interim plant, AR, full scale plant

* Identified biodegrable PCB and non-PCB capacitor dielectric

  Initial attempts for emulsified biphenyl failed

* DPO process, heat transfer conversions - Goodyear success

  Initiated process work o-PP/p-PP

  Contract work
  NASA - Lubricants and hydraulic fluids for space shuttle
  EPA - Fluids for converting heat into mechanic work - Rankine cycle engine

II. E/VCl Latex Binders

Paper Applications

Pigment Binder

E/VCl trialed on commercial equipment at Continental Can, Federal Board and Eastex

Saturation - Web and Beater

Developed formulas for wall covering at Imperial Paper

Demonstrated E/VCl in paper-nonwovens at C. H. Dexter

Functional Coatings

Clear coatings recommended at Resistane, York, Strahan

Coated cups and plates at Continental Can

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Textile Applications

Carpeting

* Helped Water Carpet increase E/VCl usage; solved backside blowing problem.
* Used E/VCl as a precoat to foam at Coronet East

  Failed on froth applications with Textile Rubber

Nonwoven and finishing

Demonstrated need for hot tensile (crosslinkable or 30°C. Tg E/VCl) at Globe

Called at Norton and Ballanger for E/VCl in abrasive and buffing wheels

New Products

* Developed Tg 30 "plastic" latex

  Initiated development of self-crosslinking E/VCl

Process

Initiated continuous process work - tube + stirred reactor

III. Paper

Surface Sizing

* Scripset at size press plus rosin at wet end boosted sizing efficiency
* Dyeing at size press plus Scripset provided new route to color paper
* Demonstrated solvent/non-solvent process for Liquid Scripsets - Montreal plant project, start-up, production
* Demonstrated Scripset 808, dry ammoniated powder - product definition, process, trials, interim production

Wet Strength

* Supported sales of Santo-Res 21/23 (1.4\overline{M}$ rate fourth quarter)
* Supported Commercial Development in obtaining FDA approval on Santo-Res 21/23
* Made new product RPC-1110-1114 for bleached tissue

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Internal Sizing

Produced Mersize LCS and efficient TFL at Seattle

Resolved poor emulsion properties of Mersize TFL

* Initiated Neuphor replacement product and process work

IV. Penta

* Developed additive package - best Penta quality-wise on market

Monitored dioxins in plant product and developed distillation purification process

* Developed process for converting by-product scrubber solutions to saleable 30% solution

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D E T A I L S

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FLUIDS

HYDRAULICS AND LUBRICANTS

L. R. Stark
P. D. Fowlkes
P. F. Heimsch
L. F. Smith

F.R. Hydraulics

Reformulated Pyrdaul E (phosphate esters) and Pydraul C (phosphate ester-hydrocarbon) fluids.

J. F. Herber
J. D. Sullivan
W. C. Warren
D. L. Kocher

Phosphate Esters

NC phosphate, a new high molecular weight non-cresylic phosphate was produced using nonylphenol (N) and cumylphenol (C) as raw materials. Cumylphenol was produced commercially for the first time. Queeny Plant achieved 99% of AR rate and yield 6 months after start-up of NC phosphate.

J. F. Herber
L. W. Bannister
R. L. Green
P. F. Pellegrin

Skydrol Fluids

Developed a Suggested Procedure for a monoepoxide stabilizer for Skydrol fluids. The monoepoxide effectively reduced hydraulic pump shaft seal leaks. Skin sensitivity is a problem. New compounds were synthesized to minimize sensitivity.

Presented paper to SAE showing superior erosion properties of Skydrol vs. competitive fluids. Skydrol shows no signs of valve erosion after 3000 hrs. of valve tests while competitive fluids from Stauffer and Chevron show signs of erosion in 100 hrs.

Provided extensive customer service of Skydrol deposits to PAA and BOAC on Skydrol 500C problems.

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L. W. Bannister
S. L. Reid
R. A. Luebke

Skylube 450, 600 and MCS 293

Continued flight tests on Skylube 450 to qualify product for airline service.

Supported manufacturing run on OS-124 for Skylube 600 and Santovac 5.

Sold test quantities of MCS 293 and reformulated MCS 293 to G.E. for CF-6 engine testing as a high AIT fluid.

R. L. Green
J. C. Wygant
R. A. Luebke

Synthetic Hydrocarbon Lubricants & Tractants

Provided Santotrac formulations to major tractive drive manufacturers, automotive industry, roller clutches and printing press manufacturers.

Developed Santotrac greases for bearings, wire rope lubricants and tractive transmissions.

Obtained favorable test results from bearing manufacturers showing 2X improved bearing fatigue life of HLD:HLP blends.

DIELECTRICS

R. H. Munch
Q. E. Thompson
S. L. Reid
R. C. Cox
D. Borowy

Capacitor Fluids

MCS 1043 (a biodegradable chlorinated biphenyl) process has been established and product has been shown to be a satisfactory capacitor dielectric impregnant with improved biodegradability but less fire resistance than Aroclor 1016.

Our program on the synthesis of new dielectric compounds, the formulation of fluids and testing of capacitors has provided the following products:

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MCS 1238, a non-halogen capacitor impregnant, had life test results equivalent to Aroclor 1016. Product is an aromatic sulfone/MIPB blend.

A capacitor dielectric impregnant containing only C, H and O gave capacitor life test results equivalent to Aroclor 1016. The product is a dimethylbenzophenone/hydrocarbon blend.

A new high 10-12 DK capacitor fluid was developed for specialty capacitors. Products are tolyl xylyl sulfone blends.

A new type of shipping drum which causes less contamination and deterioration of electrical fluid properties has been defined and tested for Aroclor 1016.

Cable Fluids

Demonstrated that H-230L could be upgraded to give the necessary electrical properties and that with a suitable inhibitor it can be kept from degrading due to oxidation.

Transformer Fluids

Initiated work to see if a high impulse strength synthetic transformer fluid could be developed. Found reason to hope that this can be done. Synthetic hydrocarbon impulses were 15 to 50% above the standard transformer oils.

PROCESS FLUIDS

J. F. Herber
Q. E. Thompson
D. R. Cova
J. C. Wygant
D. L. Kocher

Encapsulant Solvents

Santosol 100 (mono and polybenzylethylbenzene), a carbonless paper solvent for NCR, was developed by a combination of synthetic chemistry, new application testing techniques and process development. The tentative process was demonstrated in a new plant at Delaware River at design rate and product quality.

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J. F. Herber
D. L. Kocher

Dye Carriers

Failed to supply a satisfactory emulsified biphenyl, but did find out enough about evaluation of dye carriers to set up lab test procedures.

D. R. Miller
P. F. Pellegrin

Heat Transfer

Customer Conversions from Therminol FR to Therminol 66 or 55

Goodyear - Established and sold superiority of Therminol 66 over Dowtherm G.

About 100 technical service requests completed (normal year about 20).

New Fluids

Thermal stability - Some 60 materials rated on thermal stability at 660 or 720°F.

Diphenyl ether - Approvals obtained; Patent, Medical, project approved by Business Group. Process is based on phenol dehydration over thoria catalyst.

Q. E. Thompson
J. C. Wygant
D. R. Cova

New Chemicals

Started process work for the manufacture of ortho and para-phenylphenol.

CONTRACT RESEARCH

D. R. Miller
Q. E. Thompson
R. A. Luebke
H. R. Null (CED)

Environmental Protection Agency (Research Contract)

Developed specification for a Rankine Cycle Fluid for automotive engine. Recommended two fluids - a hexafluorobenzene/pentafluorobenzene blend and a 2-methylpyridine/water blend. Developed computer program to calculate entropy-enthalpy properties of non-ideal mixtures.

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D. R. Miller
R. A. Ruehrwein (proposals)
F. S. Clark (NASA)
R. L. Green (NASA)

NASA (Research Contract)

Synthesized phenylmercaptodisiloxanes for NASA contract; built a 2.4 x 10^6 DN bearing rig for NASA contract. This is an outstanding mechanical job, putting us in a class with Pratt & Whitney and SKF for high speed bearing test capability.

E/VCl LATEX BINDERS

PAPER APPLICATIONS

M. V. Merchant
M. J. Holm
Y. L. Wang
J. E. Adamove
W. G. Wagner

Pigment Binder

Demonstrated pigment binder properties on commercial equipment at Continental Can, Federal Paperboard and Eastex.

In conjunction with Eastex trial, participated in web offset print run showing E/VCl as workable pigment binder in such coatings.

Saturation: Web & Beater

Developed workable saturation formulas for wallcovering at Imperial Papers. Formulas should develop business for both E/VCl (1.2M lbs.) and Scripset 101 (45M lbs.).

Completed study for paper nonwoven at C. H. Dexter which showed E/VCl system can match acrylics for water resistance and be superior for tensile and flame retardancy.

Completed beater-add study for Chicopee Mfg. Co. Demonstrated E/VCl value for strength, hand and wet end retention. This motivated them to pilot plant runs.

Lab demonstrations show soap stripped E/VCl latexes are excellent prospects for new products. Such binders have superior water resistance.

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Functional Coatings

Recommended clear coating formulations commercially used at Resistane, York Wallpaper and Thomas Strahan Co.

Completed functional coating study for Continental Can Co. E/VCl should qualify for cup stock and paper plate coating, replacing organic solvent systems.

Established the value of E/VCl as precoat for PVDC for a high level water vapor barrier properties.

Completed FDA work needed to obtain approval under FR section 121:2526 (component of paper or paperboard in contact with aqueous and fatty foods).

Aided Riegel Paper Products in developing new flexible packaging using E/VCl as a barrier saturant.

Determined how high Tg 30°C. product may be used for barrier coating.

Characterized the heat seal and blocking properties of all E/VCl products alone and in combination with PVDC.

Wrote technical bulletin to cover market areas for Functional Coatings.

TEXTILE APPLICATIONS

M. J. Holm
Y. L. Wang
F. W. Fallwell
J. E. Adamove
E. A. Byes
W. J. Renaudette
W. G. Wagner

Nonwoven & Finishing

Did work to provide means of improving heat and light stability of E/VCl for non-durable applications.

Established for duPont the diammonium phosphate compatibility of E/VCl.

Ran commercial trial at Globe. Results gave impetus to synthesis program, showing that hot tensile strength can be obtained by self-crosslinked E/VCl.

Developed laboratory techniques to show that self-crosslinked E/VCl candidates have potential for dry cleaning and laundering requirements.

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Showed that self-crosslinked E/VCl does not undergo hydrolytic degradation upon laundering.

Made technical calls at Norton Co. and Ballanger Co. to move their use of E/VCl in abrasive and buffing wheels.

CARPETING

K. A. Pollart
J. D. Gabbard
C. J. Harris

Developed lab equipment and technique which provide meaningful results in studying carpet backing formulas. (Completed pilot trial at Monsanto-Decatur.) This aided training of product group and research application team in carpet manufacture.

Devised test to measure carpet stiffness at various temperatures. Used this to generate data on role of plasticizers and on comparison of E/VCl to SBR and blends of same for use in technical bulletins.

Provided TGA and stiffness data as needed to keep Walter Carpet on E/VCl and to increase their usage rate.

Demonstrated how backside blowing problem at Walter Carpets can be eliminated.

Assisted in the successful runs at Coronet Industries. This led to use of E/VCl as precoat to foam.

Used Coronet trials to further train product group.

Determined the relative responses, efficiencies and high shear rheologies of eleven different thickeners.

Studied accelerated aging of E/VCl vs. SBR. Technical bulletin written from data obtained.

For Textile Rubber showed that E/VCl is not adequate as frothed laminating adhesive. Yet these systems were shown to be promising for Ozite's Polygrass.

Completed 50 TSR's as requested by Marketing.

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ADHESIVES

Wrote sales bulletin outlining properties of E/VCl for use in adhesives.

Characterized the peel strengths to polypropylene of E/VCl products for use in technical bulletins.

Determined shelf life of E/VCl-amide polymers combined with reactive melamine resin. This to be used for product bulletin.

Studied plasticizer addition to E/VCl for use in product bulletin.

SPECIALTY COATINGS

M. J. Holm
Y. L. Wang
W. J. Renaudette
W. G. Wagner

Prepared films for new FDA petition for entry into can coating markets.

Prepared special metal coating for Scoville Aerosol Products. Coated units now under evaluation.

Completed trials at United Wallpaper to show that E/VCl can produce scrubbable wall covering.

Demonstrated at Columbus Coated Fabric that E/VCl is a workable binder as basecoat and printcoat for fabric wall covering. Lower price of parent company PVC kept us from business.

Determined compatibility of E/VCl for alcohol used in inks at Equitable Bag. Switched customer to use of hydrolyzed E/VCl.

Wrote technical bulletin on safe solvents for clean-up of E/VCl at Equitable Bag. Information has general interest.

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POLYMER CHARACTERIZATION

M. J. Holm
Y. L. Wang
W. F. Fallwell
J. E. Adamove
E. A. Byes
W. J. Renaudette
W. G. Wagner

Supported synthesis effort to develop new products by characterizing base polymer properties.

Developed new techniques for testing physical properties of bulk polymers. Used to characterize E/VCl and competitive products. Improved method of determining Tg.

Characterized the cure response of E/VCl and various competitive products.

Established the flammability character of E/VCl from films and filled system by LOI method and determined effects of plasticizers on flammability properties and screened FR additives.

Determined long term (1 year) aging properties of E/VCl films as measured by brightness change.

Completed studies on methods and economics for making new E/VCl products by soap stripping or deionizing.

Aided in training Marketing group in properties and applications of E/VCl through field trips, telephone calls and their visits to St. Louis.

E/VCl POLYMERIZATION

M. E. Gibbs
H. M. Andersen
S. M. Balaban
W. J. Raiff
A. J. Bindbeutel

High Strength E/VCl

Successfully demonstrated and produced a 30°C. Tg E/VCl, a high tensile, non-blocking product. Resolved tendency for higher grit and coagulum formation in 85% vinyl chloride content systems (30°C. Tg process).

Initiated interest in dry powder for special coating applications by preparation and trial of 30° Tg fine powder samples.

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Improved FR Rubbers for Textile Applications

Established feasibility of ethylene-vinyl acetate/ethylene-vinyl chloride graft systems to improve low temperature properties, achieving Tg's in the range of -20°C.

Explored techniques for running new polymer systems (necessary for vinyl acetate grafts and special crosslinking monomers) at acid pH's and made progress towards plant capability of operating autoclaves at pH 5 through cathodic protection technique.

Explored crosslinking systems for E/VCl which improved hot tensile strength solvent resistance and launderability of textile and paper composites.

Continuous Process

Demonstrated initial feasibility of two stage continuous process (initiator tube plus backed mixed finishing vessel). Production rate for continuous E/VCl process is 2 to 3 times the batch rate, depending upon composition of polymer being made.

Demonstrated monomer recycle in continuous process venting only dissolved monomer in polymer, increasing monomer yield by 10% and decreasing waste monomer incineration requirements by 70%.

PAPER CHEMICALS

SCRIPSETS

D. R. Dill
F. T. Marshall
R. W. Sanders
W. F. Vogelpohl

Two developments, Liquid Scripsets and Dry Ammoniated Scripsets, have kept us in surface sizing leadership by correcting product deficiencies, becoming environmentally more compatible and forming the cornerstone to our entry into the European paper chemical specialties market.

Liquid Scripsets

Developed Liquid Scripset 520 (RPC-1022) process via solvent-non-solvent route and demonstrated and established production at Montreal plant site.

Developed Liquid Scripset 500 process via S/NS and demonstrated at Montreal plant site. Provided products equivalent to competitive American Cyanamid product, Cypres 48.

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Developed S/NS SMA technology further with wide range (specific viscosity 1% in DMF 0.4-12.0) of molecular weights. One high viscosity model demonstrated in pilot equipment.

Dry Ammoniated Scripset

Developed two routes for a dry ammoniated maleic anhydride copolymer resin. Demonstrated feasibility in pilot equipment for liquid ammonia process (RPC-1016, Scripset 808).

Developed application technology, trialing and full commercialization within one year period for this new dry ammoniated Scripset 808.

Developed replacement product for our major Scripset 530 customer eliminating objectionable ammonia makedown and supplied material for customer trialing (RPC-1027).

Scripset Sizing

Field demonstrated two significant application studies on surface sizing:

Related basesheet variables and machine speed to size press pick-up control.

Found that interaction of internal (Mersize) and surface sizes (Scripset) will provide the optimum in performance from the customer's sizing dollar.

P&P Div., improved polypress operation fewer blown cakes in late 1972; made "high" molecular weight polymer by mass process at 35°C.; may provide route to polypress operation for higher molecular weight products.

New Scripsets

In cooperation with the wet-end group we developed an optimum rosin size (RPC-609) for the size press to be used as a fourth generation candidate.

Based on the RPC-609 technology, two 3rd generation surface sizes (RPC-1023, 1025) based on blends of RPC-609 and SMA's were developed, evaluated on pilot machine and prepared for full machine trial.

New Mill Process for Coloring Paper

Developed a new mill process for colored paper by dyeing at the size press and sizing with Scripset.

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Non-Polar Holdout

Developed back-up candidates for PCW-10 (>2.0M lbs./yr. current sales) and demonstrated performance on Weyerhaeuser pilot coater clearing way for full machine trial (RPC-117).

Utilized film forming properties of partially neutralized sodium salts of current SMA products to develop two new products currently being sampled to the non-polar holdout market.

Defoamers - New Product Line

Selected, trialled and sold Limit 33, a defoamer for Scripset. This is a new product line.

New Projects

Evaluated but presently declined the opportunity to market a fluorochemical candidate product from Allied Chemical Co.

Initiated efforts to work with H&P and CRD to develop technology utilizing propionic acid as a candidate for wood chip preservation.

Initiated a minimal effort on permanent fire retardants for paper in an effort to meet our customers' growing needs with existing greater Monsanto technology.

Marketing

Completed 90 technical service requests for specific customers. Assisted Marketing with certain customer presentations and field trials.

MERSIZES

A. J. Lauck
J. P. Petrovich
D. N. Van Eenam
C. H. Danett

Wet Strength

Developed, produced and trialed a new wet strength resin/NaOH activator system for the $20M sanitary paper market. Expect initial sales first quarter 1973 for the RPC-1110-1114 series. Produced 20M lbs. at Baxley for trials.

Supported sales of Santo-Res 21 and 23. Fourth quarter sales rate = $1.4M/yr. Have problems with 20% loss in efficiency - possibly due to changes in storage or by-product "HMD batch amine residue" from Monsanto Decatur.

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Retention Aid

Demonstrated Santo-Res 21 as a retention aid for Neuphor 100 at American Can, replacing Kymene. Advantages of Santo-Res are lower foaming, better efficiency than Kymene (Hercules' product).

A. J. Lauck
M. H. Berry
O. E. Mueller

Sizing - Mersize

Produced Mersize LCS at Seattle to capture West Coast business. Monitored and advised in plant quality of Mersize TFL, maintained uniform high efficiency, resolved poor emulsion characteristics. Supported Marketing customer service - 50 requests.

Developed optimum liquid rosin size (RPC-609) for surface size application and produced initial batch at Baxley.

Helped develop interaction internal/surface sizing concepts now being used to promote our Scripset line.

A. J. Lauck
J. T. Daust
E. D. Clemons (CED)
M. H. Berry

Dispersed Rosin Size

Have shown limited feasibility procedures for both a solvent and molten route to a dispersed rosin size. The lower manufacture and capital cost molten method will be developed to a Tentative Process and engineering design stage in early 1973.

PENTACHLOROPHENOL

D. R. Dill
S. H. Vogel (DKL)
D. R. Cova
J. E. White

Completed plant optimization of additive package which provides Monsanto with the best current commercial pentachlorophenol.

Developed processes for converting by-product scrubber solutions to a saleable 30% Santobrite solution.

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Low dioxin Penta - Completed a tentative process for distillation route; will evaluate a "cleaner chlorination" and crystallization route. Dow registered a "clean" Penta in late 1972.

Monitored dioxins in our standard commercial production and in samples from all laboratory studies.

Selected and demonstrated a superior oil for coating Penta prills to minimize sublimation.

Demonstrated biodegradability of pure pentachlorophenol by outside contract at Purdue University in cooperation with other Penta producers. Found impure commercial Penta is more toxic to bugs.

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