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SSME Main Combustion Chamber (MCC) "Hot Oil" Dewaxing

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Abstract

In an attempt to comply with the changing environmental regulations, a process was developed for the replacement of perchloroethylene in the dewaxing of the Space Shuttle Main Engine (SSME) Main Combustion Chamber (MCC) and other associated hardware filled with the Rigidax® casting compound. Rigidax® is a hard blue-dyed, calcium carbonate filled thermoplastic casting compound (melting point 77° C) that is melted and poured into hardware cavities to prevent contamination during material removal processes, i.e. machining, grinding, drilling, and deburring. Additionally, it serves as a maskant for designated areas during electroforming processes.

Laboratory testing was conducted to evaluate seven alternate fluids for the replacement of perchloroethylene in the dewaxing process. Based upon successful laboratory results, a mineral oil was selected for testing on actual hardware. The final process developed involves simultaneous immersion and flushing of the MCC channels using a distinct eight stage process. A nonvolatile hydrocarbon analysis of a solvent flush sample is performed to determine the hardware cleanliness for comparison to the previous perchloroethylene dewaxing process.

Introduction

The Space Shuttle Main Engine (SSME) Main Combustion Chamber (MCC) is a cylindrical, regeneratively cooled, structural chamber consisting of a coolant liner, a high strength structural jacket, coolant inlet and outlet manifolds, and a throat shell (see Figure 1a). The MCC contains the burning propellants with chamber cooling provided by hydrogen flowing through the coolant channels in the liner.

The liner is spin forged Narloy Z which is machined to the same contour as the inner diameter of the structural jacket. The outer surface of the liner (Figure 1b) has 390 milled axial coolant channels measuring approximately 24" linearly (from the aft to the forward end) with varying rectangular cross sectional dimensions ranging from 0.035" x 0.093" at the throat to 0.060" x 0.247" at the aft end of the liner. After the milling operation the channels are deburred, inspected and then readied for application of a wax maskant. The wax used is Rigidax®, a calcium carbonate filled thermoplastic compound with a melting point of 77°C. This particular compound is used because it is dimensionally stable when processed through the plating solutions.

Molten wax is poured into each of the 390 channels while the liner is continuously rotated horizontally. After cooling to room temperature, the liner is wet sanded by hand to expose the Narloy Z on the lands between the channels; and maintain a 32 surface finish. The required amount of copper (Cu) layer is then electrodeposited onto the liner to closeout the channels and to serve as a hydrogen embrittlement barrier followed by electrodeposition of the required amount of nickel layer. The electrodeposited nickel (EDNi) is then machined to the desired dimensional requirements. The dewaxing operation to remove the Rigidax® in the channels is performed following this step.

Previously, the dewaxing operation was performed by a combination of vapor degreasing and channel flushing with perchloroethylene following the process flow as shown in Figure 2. The chamber was then filled with perchloroethylene and allowed to soak for thirty minutes while suspended in the vapor zone of the degreaser after which the solvent was collected for verification analysis. The collected sample was evaporated and the residue analyzed for wax content using infrared spectrophotometry (IR). The cleanliness requirement is less than 0.50mg wax/200ml effluent. Following a drying step, an acetic acid and deionized water flush was performed to remove any residual calcium carbonate that may have been present after dewaxing. A final drying was performed, followed by borescope inspection viewing to inspect for any blocked channels.

Rocketdyne initiated an environmental task to eliminate the use of perchloroethylene for this operation due to the listing of perchloroethylene as a suspected carcinogen in California's list of air toxics, its regulation by rule AB2588 (Comprehensive Community Notification) and Proposition 65, and the inability to meet the stringent Occupational Safety & Health Administration (OSHA) allowable exposure limits (AEL) of 25 ppm. The focus of the replacement task was to develop a process that would be environmentally compliant, compatible with the hardware substrate materials, and yield cleanliness levels comparable to those achieved with the perchloroethylene process. To minimize the cost and time associated with extensive hardware trials, laboratory screening tests were performed to select the most feasible candidates followed by process scale-up.

Procedure

Identification Of Candidate Cleaners

Initial efforts focused on a literature review, an industry survey of Rigidax® users, supplier surveys, and recommendations by the in-house Environmental and Health & Safety departments. A list of candidate cleaners was compiled and the cleaners were reviewed for physical and chemical properties, cleaning effectiveness, material compatibility and integration into the existing manufacturing processes. Seven alternate fluids, as listed in Table 1, were evaluated during the laboratory test phase.

Laboratory Test - Dewaxing Effectiveness

Laboratory wax removal tests were performed using a 3" x 3" cut away segment of a scrap MCC. The test segments were pre-cleaned by vapor degreasing with perchloroethylene, weighed, filled with Rigidax® and reweighed. A 450-550ml sample of the test fluid was placed into a 600ml Pyrex beaker. The fluid was heated using a hot plate stirrer to the desired test temperature range as indicated in Figure 4. The Rigidax® filled segment was suspended in the test fluid which was agitated using a Teflon coated magnetic stir bar. The segment was periodically removed and visually inspected for wax removal. The time required to open the channels and remove the wax was recorded. When the MCC segment was visually clean, it was cooled to ambient temperature and reweighed to verify wax removal. If residual test fluid remained on the test segment, it was rinsed in hot deionized water at 60°C for one hour. The MCC test segment was then reweighed to determine any weight gain or loss. The same procedure was used for all of the fluids evaluated except as noted.

In the case of the hot oils (DXE and Turco 6802), an aqueous cleaning step was added prior to the deionized water rinse. The hot oil test required an additional alkaline cleaning step to remove the oil after dewaxing while the other fluids were tested as stand alone dewaxing fluids. Only rinsing was required with the Axarel and Brulin, and only evaporative drying was required with the KNI-2000 solvent and the kerosene.

Laboratory Test - Material Compatibility

General corrosion testing was performed with the Turco 6802 oil and Dupont Axarel 56 to determine the compatibility of these fluids with the hardware alloys. Five specimens each of nine metals were evaluated. The test panels included:

- Nickel Alloy 718
- Nickel Alloy 625
- Iron Nickel Alloy 903
- EDNi
- 21-6-9 Stainless steel
- A-286 Stainless steel
- Hastelloy® W
- Copper
- Narloy Z

Each panel was cleaned with Scotch-brite and deionized (DI) water to a water break-free surface, dried and weighed. One specimen of each alloy was set aside as a control. From each alloy set one panel was immersed in Axarel 56 at 49-54°C, one in Axarel 56 at 79-85°C, one in the Turco oil 6802 at 79-85°C and one in the Turco oil at 99-121°C for twenty minutes. The panels were allowed to stand in the cooling fluid for 24 hours. Upon removal from the fluids, the panels were lightly flushed with DI water; followed by a four minute immersion rinse in DI water. The panels were oven dried for one hour at 91-96°C. The panels were reweighed upon cooling and then evaluated visually and by use of a 30-100X microscope for any noticeable corrosion.

Pilot - Scale Tests

A pilot plant was assembled to allow testing of a full-size MCC. The final configuration of the pilot plant consisted of the following:

- A 4ft x 4ft x 4ft stainless steel tank containing the Turco 6802. The tank was equipped with a centrifugal filtration unit to filter out the calcium salt from the oil, and a 100 micron size rated cartridge filter to prevent particulate matter from entering the MCC channels.

- A 4ft x 4ft x 4ft stainless steel tank containing the 75/25 Axarel 56/water. The tank was equipped with a 100 micron size rated cartridge filter, two 15KW Chromalox external heating elements with safety interlocks to protect against any overheating. Agitation manifolds were added to ensure effective emulsification of the Axarel/water mixture. The agitation manifold consisted of four nozzles in each corner at the bottom of the tank.

- Two 4ft x 4ft x 4ft polypropylene tanks, one containing the Turco Vitroklene alkaline cleaner and the second containing acetic acid. Both tanks were equipped with 30 micron size rated cartridge filters.

- One 4ft x 4ft x 4ft polypropylene lined tank for intermediate water flushing and

- One stainless steel tank measuring approximately 4ft x 4ft x 4ft utilized for final rinsing with deionized water which was equipped with a 20 micron size cartridge filter.

Each tank was equipped with a 5hp magnetically coupled pump except the acetic acid tank which was equipped with a 1 1/2hp pump used at variable flow rates of 30-50gpm. Each pump was equipped with a safety interlock to shut down the pump if a loss of prime condition should occur. This ensures that the pumps do not run dry. Each tank was equipped with two 15KW immersion heaters except the Axarel as noted.

Testing in the pilot facility was conducted using a full-size scrap MCC (Figure 1). The MCC was first pre-cleaned by vapor degreasing with perchloroethylene and then filled with Rigidax®. Prior to wax application the MCC was heated to 177°C. Melted wax was then poured at 149°C into the channels from the forward end and allowed to gravity flow to the aft end. Once filled, the chamber was allowed to cool to room temperature. Dewaxing was initiated by first placing the MCC in an oven heated to 135-163°C for 2 ± 0.5 hours to remove the bulk wax as per the normal dewaxing process. The remaining wax was then removed using the experimental hot oil dewaxing flow process as shown in Figure 3.

Each cleaning stage involved an initial soak period by immersing the MCC in the fluid and filling the internal cavities after which the fluid was flushed through the channels (from the aft end up to allow observation of the effluent fluid). Gaseous nitrogen (GN2) was simultaneously injected into the fluid stream to enhance a scrubbing action in loosening and dissolving wax from the channel walls. GN2 was used with all the flushing operations except the acid flush. Process optimization including times, temperature, fluid flow rates and GN2 flow rate as well as upgrading of the 1/2 to 3/4 hp pumps to 5 hp pumps were performed as testing progressed. Verification for the effectiveness of wax removal, was performed using TCA (1,1,1, trichloroethane). The MCC was filled with TCA and allowed to soak for 30 minutes at 66°C. The TCA was collected and the wax content determined by IR spectrophotometry after solvent evaporation.

<u>**Production - Scale Testing**</u>

Production scale testing was conducted primarily using the scrap MCC and the dewaxing flow process (Figure 3) with implementation of the optimized parameters. Several tests were conducted to evaluate the repeatability of the process. Process specification approval from the customer was obtained and facility modifications were completed to permit use of the process on production hardware. Production hardware continues to be dewaxed using the hot oil process.

Results & Discussion

<u>**Laboratory Rigidax Removal**</u>

Laboratory test results are presented as the amount of time, at a particular fluid temperature, required to remove the wax from the channels of the test segment (Figure 4). NVR evaluation was not necessary since the test segment channels were visually inspectable for any blockages or residual film on the channel walls. The results from the initial laboratory experimentation using the DXE oil showed that the test fluid was capable of dewaxing the MCC test segments based upon the selected time and temperature parameters. Following the completion of seven successful laboratory tests, a primary process set up consisting of the DXE hot oil dewax, alkaline clean, acid flush, DI water rinse, dry, and a TCA verification was proposed for trial at the pilot scale testing.

<u>**Pilot & Production Scale Rigidax Removal**</u>

Test results at the pilot & production scale levels are presented as wax content of the TCA solvent sample obtained from the MCC subsequent to the cleaning cycle. The results are classified as pilot-scale and production-scale. The pilot-scale refers to the original plant with minimal processing capability and the production-scale refers to the upgraded facility with the more robust processing capability. NVR hydrocarbon analysis was used since the channels are not visually accessible. Results from both the pilot and production scale tests are graphically represented on Figure 5.

In the pilot-scale testing, it was determined that the primary process was inadequate in the dewaxing of the MCC. Additionally, the DXE oil vapor was offensive to the employees. Furthermore, each process step lasted 8 hours, requiring one week to completely dewax an MCC. This process also lacked intermediate water flushing, resulting in severe contamination of the process tanks (from excessive carry-over) and subsequent incomplete cleaning. Test A (Figure 5), was performed using perchloroethylene. Test B, in Figure 5 is the average of five tests performed with the DXE process.

A complete re-evaluation of the dewaxing process which included evaluating test fluids other than the DXE oil was performed (Figure 4). Turco 6802 oil was selected at the conclusion of the laboratory testing. Turco 6802 contains a surfactant and with its improved rinsability exhibited a higher potential for effective dewaxing. Both the Axarel 32 and 56 showed effective dewaxing potential but the low flash points (93-99°C) diminished their viability as substitutes. The KNI-2000 solvent, kerosene and Brulin Exp. 492-1 were ineffective and no further tests were conducted with these fluids.

Use of the Turco 6802 oil to replace the DXE in the preliminary process set eliminated the odor encountered with the DXE but the parts still failed to meet the NVR requirement. The results showed the oil effectively dewaxed the MCC but the alkaline cleaner (Turco 3878) could not effectively remove the residual oil/wax mixture from the liner walls. Vitroklene cleaner was substituted for the Turco 3878 due to its higher operating temperature but did not enhance the cleaning effectiveness either. It was critical that all processing fluids in the dewaxing process were operated above the melting point of the Rigidax® to avoid any form of re-solidification of the wax.

The inability to meet the NVR requirement resulted in evaluating the use of GN2 during the flushing process and an Axarel 56 closed loop soak and flush step at the end of the hot oil flush. The GN2 was intended to enhance the scrubbing action during flushing and the Axarel 56, being a mixture of aliphatic hydrocarbons, aliphatic esters, and a nonionic surfactant, would effectively remove the residual oil/wax mixture and followed by an alkaline clean (Vitroklene). This combination; Turco 6802 oil → Axarel 56 → Alkaline clean(Vitroklene) → Acetic acid → DI water rinse → TCA verification, finally yielded passing NVR results (Figure 5, test C) and thus provided the basis for a permanent replacement for perchloroethylene dewaxing process.

Due to the low flash point of the Axarel 56, follow - up laboratory tests were designed to test the Axarel in an emulsion concentration at various temperatures in an attempt to eliminate the hazard. The tested emulsion ratios were effective in dewaxing the MCC test segments and the fluids would not flash when tested. Testing following the addition of the Axarel in the dewaxing process improved the potential for yielding passing cleanliness results (Figure 5) thus proving the process production ready. Pilot tests D, E and G were also conducted with test C combination but did not pass the cleanliness requirement test due to parameter changes. In tests D and E, the operating temperature of the Axarel was lowered and in test G, a shortened processing time for the overall process was used. Tests F and H were conducted with combination C parameter and both yielded passing results. Production scale tests that followed (Tests 1 through 5, Figure 5) were conducted on a continuous basis (using the same parameters as test C but with the 75/25 Axarel/water emulsion) to collect data and determine the repeatability of the process. Results show consistency in meeting the cleanliness requirement and so have the production hardware processed to date.

The following events occur during the dewaxing process. The hot oil first dissolves the wax while the calcium carbonate filler material drops out of solution. The Axarel, in turn, dissolves the residual oil/wax film left on the channel walls and an aqueous alkaline cleaner then removes the Axarel. The acid flush eliminates any residual calcium carbonate salts that may be left in the channels. A final DI water flush is performed to remove any cleaning solution residues.

Material Compatibility

All the tested alloys exhibited compatibility with the tested fluids. There were no evidence of surface corrosion or intergranular attack (IGA) when the coupons were examined visually or using a 30X to 100X microscope.

Conclusion

It can be concluded from the extensive testing performed thus far that:

- The hot oil dewaxing process is a technically acceptable process for production implementation and is a viable replacement for perchloroethylene dewaxing.
- The Axarel emulsion eliminated flash point concerns.

Acknowledgment

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TEST FLUIDS	MANUFACTURER	FLASH POINT	BOILING POINT
DXE ($C_{18}H_{22}$) Dixylylethane	AllCo Chemical Corporation	163°C (325°F)	335°C (635°F)
Re-entry-KNI Solvent 2000	Envirosol Inc.	64° C (148°F)	190-215°C (375-420 °F)
Turco 6802 Oil - Petroleum distillate w/Octylphenoxypoly (Ethyleneoxy) Ethanol Surfactant	Turco Products Inc.	149°C (300 °F)	260°C (500 °F)
Axarel 32	Du Pont Company	94-99°C (200- 210° F)	221-295°C (430-563°F)
Axarel 56	Du Pont Company	94-99° C (200-210°F)	216-260°C (420-500°F)
Kerosene	J.T. Baker	65-85°C (150-185°F)	79-163°C (175-325°F)
Brulin Exp 492-1	The Brulin Corporation	N/A	N/A

Table 1: Description of Test Fluid

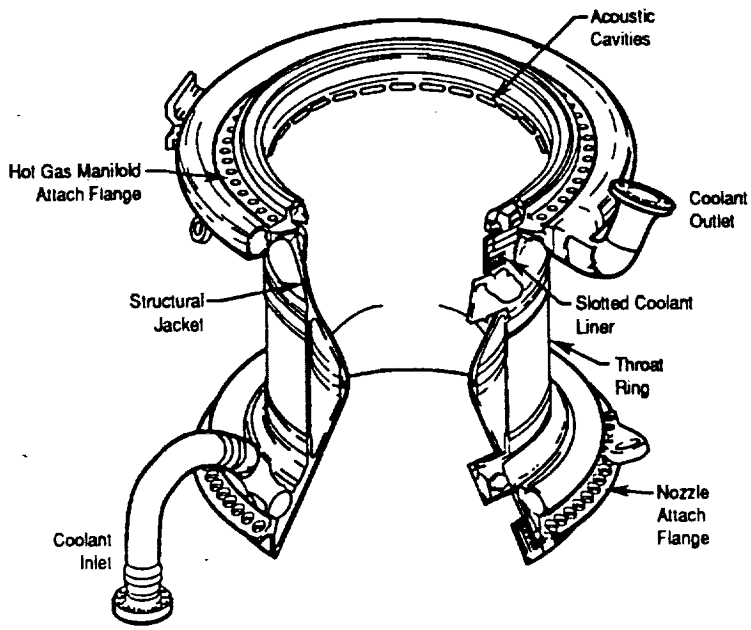

Figure 1a: Main Combustion Chamber

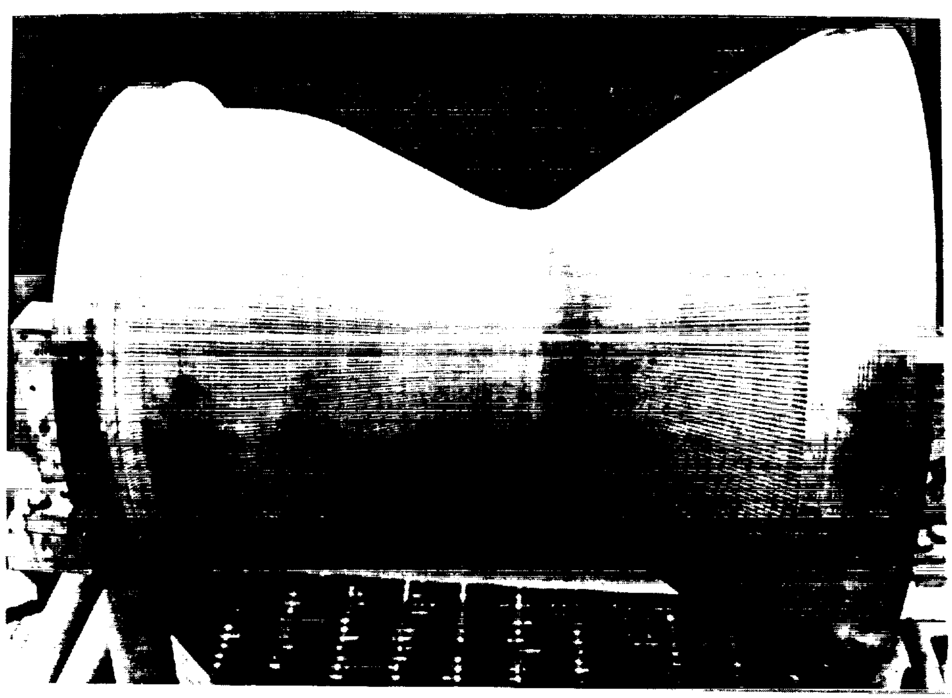

Figure 1b: MCC Liner With Slotted Channels

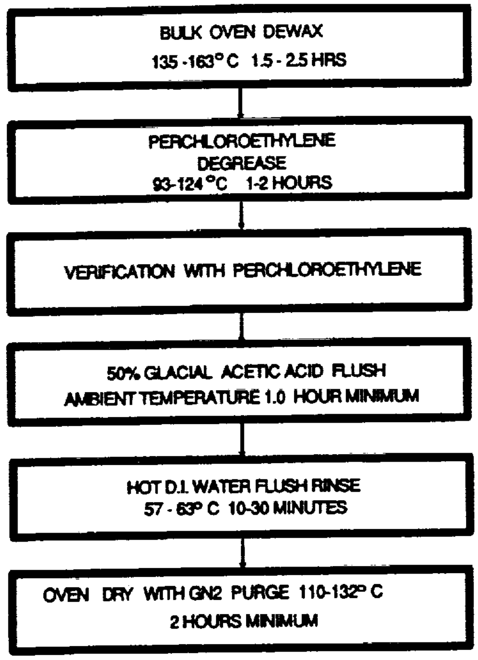

Figure 2: Perchloroethylene Dewaxing Process

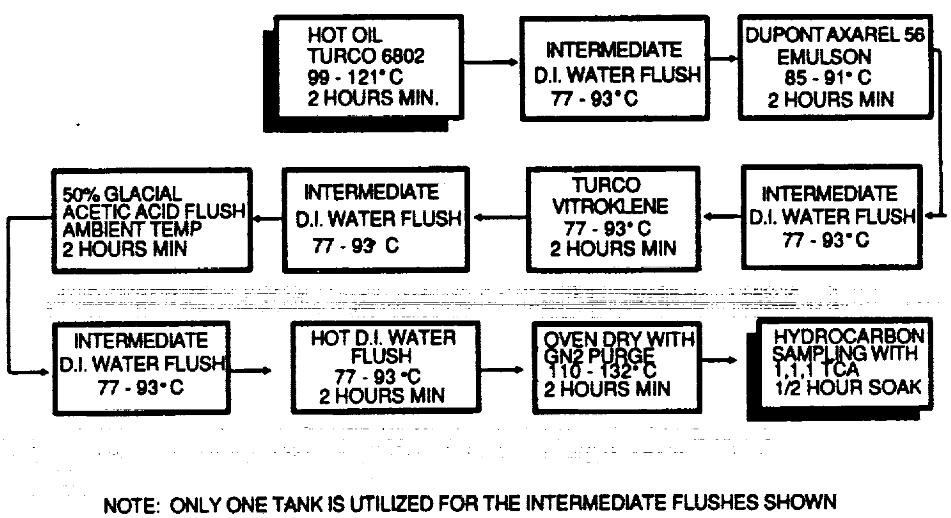

Figure 3: MCC Dewaxing Flow Process

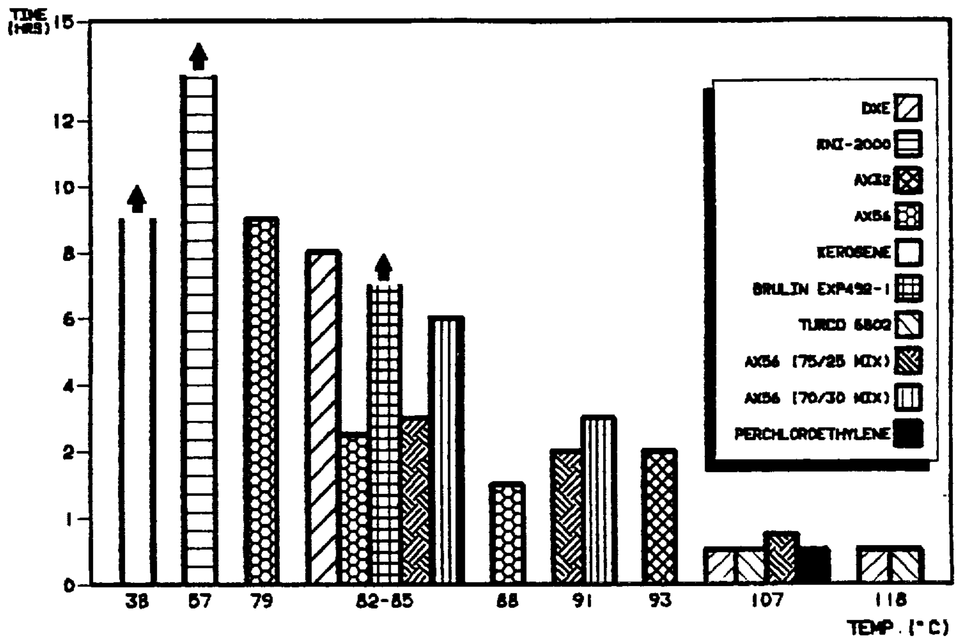

Figure 4: Dewaxing Time And Temperature

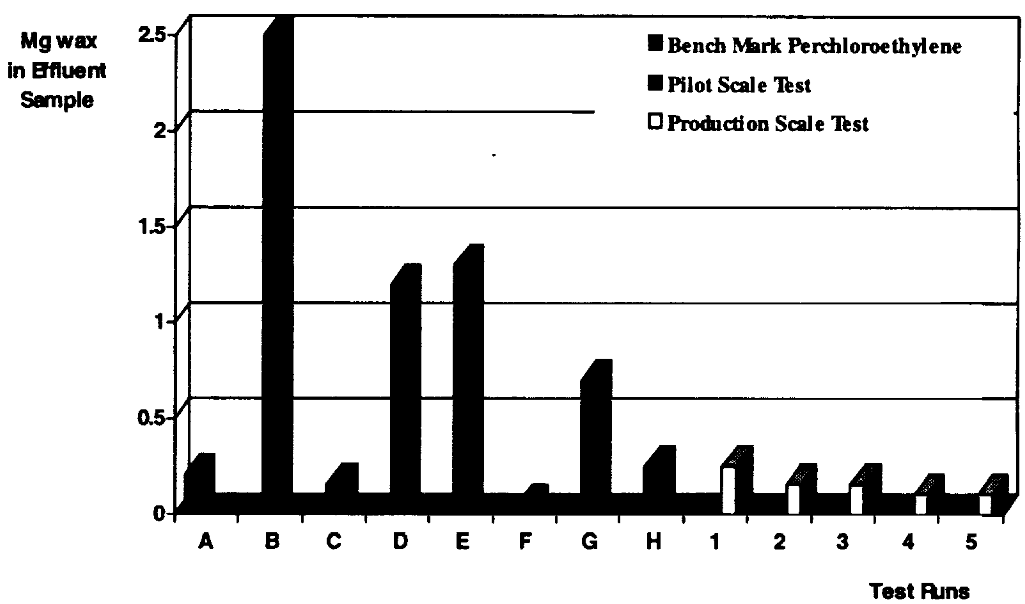

Figure 5:Results from Pilot and Production Scale Tests