CONTINUOUS CARBON FILTRATION OF NICKEL PLATING BATHS

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Introduction

Recent developments which immobilize the activated carbon within the filter media produce better solution flow rates while exposing more of the adsorbent surface of the activated carbon for more efficient removal of organic contaminants from bright electrolytic nickel plating solutions.

Electrokinetically bonded carbon is compared to powdered activated carbon by measuring the differences in hydraulic permeability and adsorptive properties. The usage and management of these two types of carbons as a precoat for bright electrolytic nickel bath filters are discussed along with waste minimization and convenience.

Background

Bright electrolytic nickel plating baths must be filtered for two reasons: First, to remove insoluble particles from the plating bath. Failure to remove these particles will result in unacceptable rough plating. Second, to remove the organic contamination from nickel brightener breakdown and oil drag-in. Failure to remove these and other organic contaminants will result in dull nickel deposit and(or) pitting. Continuous carbon filtration is the most efficient and reliable method to assure consistent roughness-free quality bright electrolytic nickel plating.

Suppliers of filters and manufacturers of electrolytic nickel brighteners recommend a turnover of the solution one (1) to three (3) times per hour, through a media removing the insoluble particles and organic contaminants. It is the author's experience that most large nickel plating baths use a filter which is precoated with a filter media for removal of insoluble particles, and powdered activated carbon (PAC) for removal of soluble organic compounds. The filter media is usually diatomaceous earth (DE), cellulose fiber, or a combination of both. The media is mixed in a slurry tank and fed into the filter to form a uniform precoat on the filter leaves. PAC is then mixed in the filter slurry tank and fed into the filter to form a second coating over the filter media. The filter media must be applied first to prevent bleed through of the fine PAC particles into the plating bath. It is well known that activated carbon tends to form densely packed beds, particularly in it's finely divided state where it is most efficient. Therefore, it is difficult to efficiently utilize PAC for the continuous filtration of nickel solutions.

Mechanisms of Filtration

There are three fundamental mechanisms by which particles are filtered from liquids: Surface straining, depth straining, and adsorptive filtration[1], Fig 1. Surface and depth straining occur

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when the interstitial pores of the filter media are smaller than the particles to be filtered. Straining is an efficient method; however as the pores clog, there is a high rise in pressure drop across the filter reducing solution flow and turnover rate of filtration. Surface and depth straining are the filter mechanisms of PAC, DE or cellulose fibers. The consequence is frequent maintenance and shut down of the filter to replace the PAC and media. Adsorptive filter media have extremely large pores, resulting in very little straining of particles. Adsorptive filtration occurs when a contact is made between a particle on or in the filter media; the particles then adhere to the filter media by electrostatic attraction or Van der Walls forces. Therefore, an adsorptive filtering media with PAC will provide excellent solution flow while removing insoluble particles and organic contaminants. A product has been developed and is successfully being used by many bright electrolytic nickel platers.

New Developments

A patented[2] new precoat media, consisting of electrokinetically bonded carbon (EBC)[3], is a homogeneous mixture of powdered activated carbon and fibrous materials. The EBC filter media is not a simple mixture of its ingredients. The ingredients have been interacted effectively to avoid segregation and migration to minimize pressure drop and eliminate binding of filter elements. Since more surface area of the carbon content is in contact with the solution flow, there is faster and better adsorption of the soluble organic breakdown products from the nickel brighteners and oil drag-in. Also, EBC is semi-moist in order to maintain the electrokinetic bond of carbon to the cellulose fibers, and therefore, the product is dust free. Users of EBC (frequently referred to in the industry as "carbon on a rope"), have reported a preference for this single, dust free media which provides faster organic removal with better solution flow and less frequent clean out of the filters.

Although the final decision on the effectiveness and advantages of a product is its success in production, the following laboratory experiments were performed to verify the results obtained by users of EBC in production.

Laboratory Results (Physical Properties)

The hydraulic permeability of a precoat material is one of the important properties in filter applications. If the solution flow through the filter precoat is restricted, the rate of turnover will be reduced, and the filtration will be inefficient. Nickel baths contain suspended particles which must be removed along with the dissolved organic. Typically, filters are run at a constant flow rate to a pressure endpoint. A precoat material with a high hydraulic permeability minimizes the initial pressure differential across the filter and maximizes the volume of bath which can be treated per precoat. Test results[4] have shown that the hydraulic permeability of EBC is much greater than that of PAC.

These tests were performed at 20°C (68°F) on a pressurized precoat filtration apparatus shown schematically in Fig. 3. Hydraulic permeability is expressed in terms of the Darcy unit, defined as follows: A material having a permeability of one Darcy unit passes one milliliter per second per square centimeter of liquid having a viscosity of one centipoise through a cake of one centimeter thickness at a pressure differential of one atmosphere. Individual tests were performed with EBC, PAC, DE and various mixtures, and the results are given in Table I.

PRODUCT	DARCY
Electrokinetically Bonded Carbon (EBC)	1 to 1.5+
Fine DE	0.08
Medium DE	0.3 to 0.5
Medium Coarse DE	1.0
Coarse DE	3.0
Powdered Activated Carbon (PAC)	0.1
Fine DE + PAC, 1:1	0.1
Medium DE + PAC, 1:1	0.1
Med Coarse DE + PAC, 1:1	0.4
Coarse DE + PAC	0.4

TABLE 1

As shown the hydraulic permeability of EBC is 10 to 15 times that of PAC alone, or with fine and medium DE.

Although good solution flow rates and more turnovers are important, the filter media must be efficient in removing suspended solids. To determine the efficiency in removing suspended solids, a test was performed on a small precoat filter pilot plant. The test conditions included precoating a cylindrical septum with EBC at a dosage of 977 dry g/m^2 (3 oz/ft^2). The influent area flow rate was 9.8 m/hr (3 ft/hr)., into which iron oxide (Fe_2O3_3), was injected to achieve an influent concentration of 0.5 mg/L. Over the course of the run, the differential pressure across the filter was measured and recorded and effluent samples were drawn periodical through 0.45 micron Millipore filters to detect any iron or precoat leakage into the effluent. The data indicated a holding capacity of about 0.28 g Fe_2O_3/g (4 oz/lb) EBC to differential pressure end point of 1.0 kg/cm^2 (14.2 psi) Fig. 2. During the run, no leakage of iron or precoat material was detected on the effluent Millipore filters, indicating virtually 100% removal efficiency of iron oxide. In addition the integrity of the precoat was maintained. The same test with diatomaceous earth, indicated EBC had a holding capacity several times greater.

This efficiency of removal was verified on a commercial 160 sq. ft., 8,000 gph, vertical leaf filter. After the filter had been cleaned and precoated with 40 pounds of EBC from the slurry tank, a cup of plain PAC was added to the slurry tank and recirculated through the EBC precoat. Samples of solution were immediately collected from the down-stream side of the filter and checked for carbon particles by passing through Whatman 40 filter paper in a glass funnel. No black particles of PAC were found on the lab filter paper.

Laboratory Results (Adsorptive Properties)

The next series of laboratory tests compared the organic removal effectiveness of EBC and PAC when filtering bright electrolytic nickel plating baths. Rather than artificially contaminating a nickel plating solution, actual production bright nickel plating baths were used for the comparison. Samples were obtained from three different production electrolytic plating baths using different brightener systems.

A. 100,000 liters (26,000 gallons) bath used for bright electrolytic nickel rack plating of wire and tubular steel fabricated parts on a return type automatic.

B. 3,800 liters (1,000 gallons) bath used for bright electrolytic nickel rack plating of tubular furniture on a hand operated hoist line.

C. 57,000 liters (15,000 gallons) bath used for bright electrolytic nickel rack plating wire and tubular steel fabricated parts on a return type automatic.

The instrument selected to measure the degree of soluble organic contamination was a UV spectrophotometer. This instrument was chosen because although Hull cell panels show when a plating bath is pitting or producing dull nickel deposit, UV scans will indicate when a bath is approaching dangerous organic contaminant levels ("a dirty bath") before they show up in production or on a Hull cell panel. Scans were made from 260 to 340 nm on a Beckman DU-50 spectrophotometer using the PEAK PICK/POINT PICK program. The points chosen were the beginning and ending scale of the spectrophotometer (260 & 340 nm) and the two points generally used to determine carrier (saccharin) concentrations (285 & 315 nm). A comparison of the absorbency of a new bath and a production bath, Graph No. 1, the used bath has a higher absorbency reading.

The three production baths were scanned prior to filtering. Using a Buchner funnel, one liter samples of each bath were filtered through either (1) EBC, or (2) PAC (over DE) three times and then subjected to a UV scan after each pass. The amount of filter media used was 0.9 kg/m^2 (3 oz/ft^2) of EBC or 0.15 kg/m^2 (0.5 oz/ft^2) of PAC over 0.9 kg/m^2 (3 oz/ft^2) of DE (EBC contains approximately one-sixth PAC. The same quantity of PAC were present in both filter precoats). For the test, the baths were kept at approximately 60°C (140°F) with flow rates maintained at 0.25 m/hr.

As shown by the UV absorbencies of all three baths indicate the EBC was more effective in removing the organic contaminants, Graphs No.1,2,3,4.

Production Case Histories

In the past four years EBC has been used in production by bright electrolytic nickel platers. One of the current plating companies using EBC for filtration of decorative nickel plating baths has seven nickel baths for a total quantity of 380,000 liters (100,000 gallons). Prior to EBC, they used 0.6-0.9 g/cm^2 (2-3 oz/ft^2) of cellulose fibers and 0.15 g/cm^2 (0.5 oz/ft^2) of PAC. Due to flow restrictions, measured by a pressure valve up-stream from the precoat, the filters were cleaned as often as twice per week. With EBC at 1.2 g/cm^2 (4 oz/ft^2) as the precoat, filters run two (2) to four (4) times longer before cleaning. (Note: The user would add PAC between clean outs which would further restrict the flow. EBC is now added which does not noticeably affect the flow)

The user estimated clean outs of each filter required approximately one hour of labor or a total of 800 man hours per year to clean eight filters twice a week. Because EBC has required less frequent clean outs, the labor time has been reduced by better than half. Where cellulose floats and takes time to wet out, and PAC is dusty and require use of respirators by operators, EBC has no dust and normally does not require respirators, making the recharging of precoats an easier task.

An additional advantage of EBC is the minimization of solid waste. EBC precoat has one-third the dry volume of conventional media and PAC. The clean out volume of precoat is substantially less. (Note: EBC as received is two-thirds moisture. The moisture is essential to maintaining the electrokinetic bond to carbon. The quantity recommended for precoating is based on it's moist weight). EBC is estimated to reduce precoat solid waste by 80-85% because of it's volume and less frequent clean outs. Since each clean out of precoat takes nickel solution with it, there is an additional 80-85% reduction in nickel solution loss and treatment.

They have used EBC for over four years and report no detrimental affects on their nickel bath chemistry.

Another case history has replaced PAC with EBC because the dustiness of PAC caused printed circuit board failure in the electronic circuit boards of hoist transfer controls.

Still another user of EBC drastically reduced the frequency of batch treatments of their electrolytic nickel plating baths to maintain good ductility and steptest readings. The quality of plating required by this automotive supplier has been maintained with an annual savings of $50-60,000 in labor cost and chemicals.

Summary

It is probably safe to say most platers couldn't care less whether their filtration is straining or adsorptive, as long as it is adequate to remove the contaminants that prevent a quality nickel deposit. However, the need to reduce cost and minimize hazardous solid waste is now on the mind of every plater. Although powdered activated carbon has been effectively used in the past, it is inefficient and inconvenient. Every plater using precoats has experienced the displeasure of handling PAC. Now there is the option of using electrokinetically bonded carbon.

EBC is truly a unique development with physical properties which are a major breakthrough in solution treatment. This technology has been used successfully in hundreds of steam electric power plants around the world for the polishing of condensates and for the removal of radioactive wastes. After introducing the technology to nickel platers, EBC has been rapidly accepted and preferred. Currently it is estimated that twenty to forty percent of the total volume of bright electrolytic nickel baths in USA are being continuously carbon treated with EBC. Laboratory evaluation has verified it's effectiveness. Industrial applications have proved EBC precoat is easy to use, reduces labor and chemical costs, while also minimizing solid waste.

REFERENCES

1. J. H. Raistrick, "The Adsorptive Filtration of Liquids", Filtration and Separation, March/April, 1981, p. 149.

2. Patent No. 4,238,334, December 9, 1980.

3. Product of Benchmark Chemical Corporation, Indianapolis, IN.

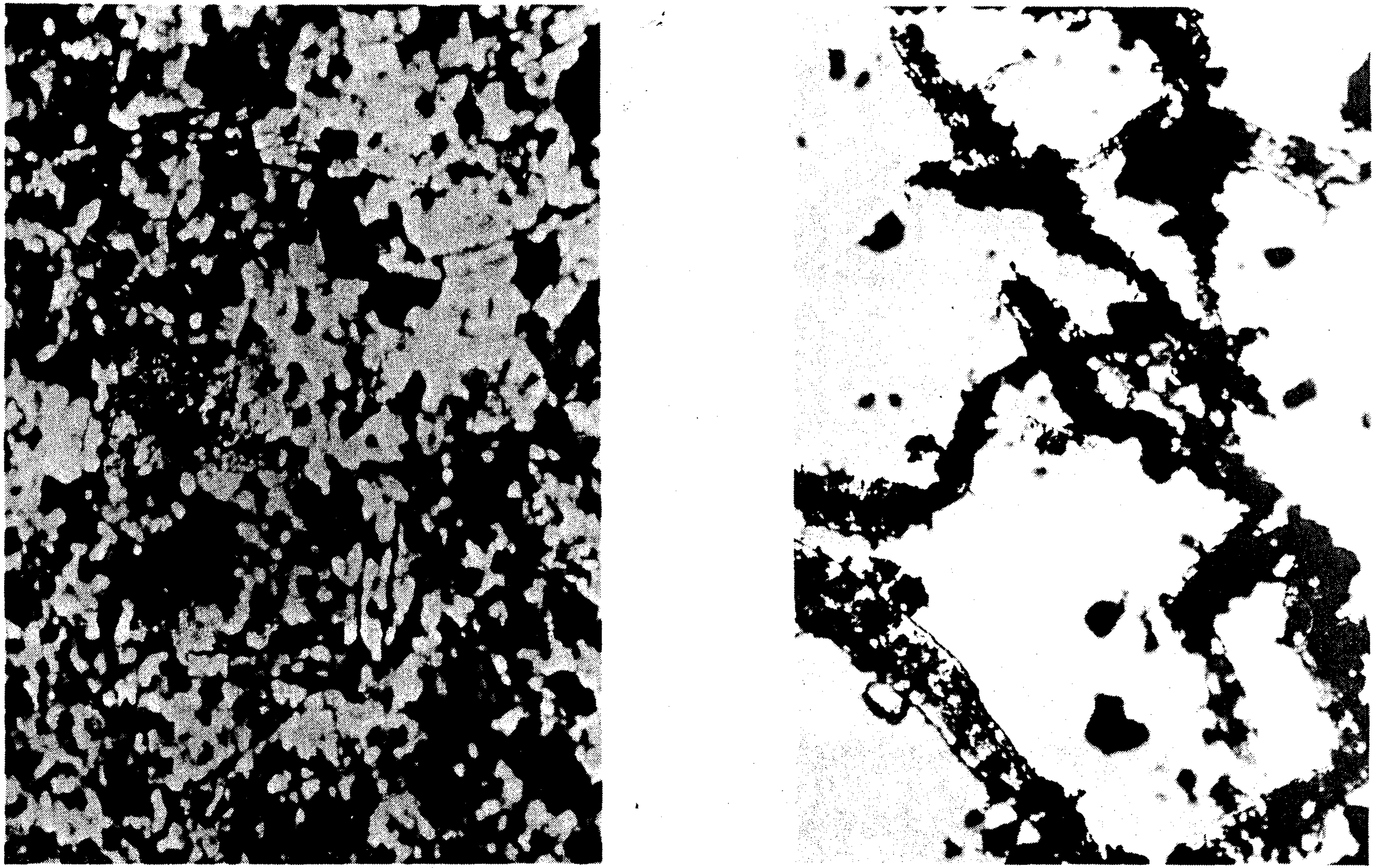
4. L. W. Brehm, M. E. Prystupa and J. F. Giannelli, paper presented at the Waste Management '83 Symposium.

Surface Straining Filtration

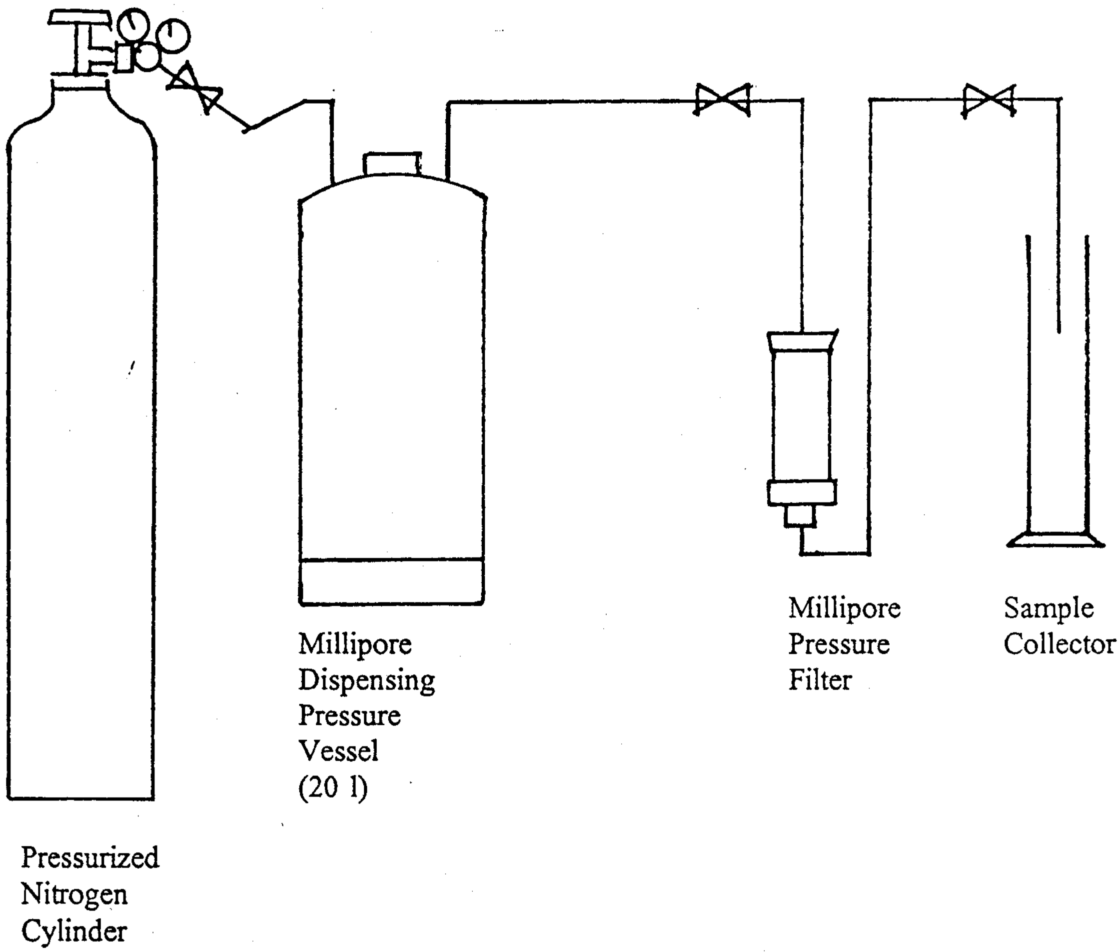
Depth Straining Filtration

Adsorptive Filtration

Figure 1

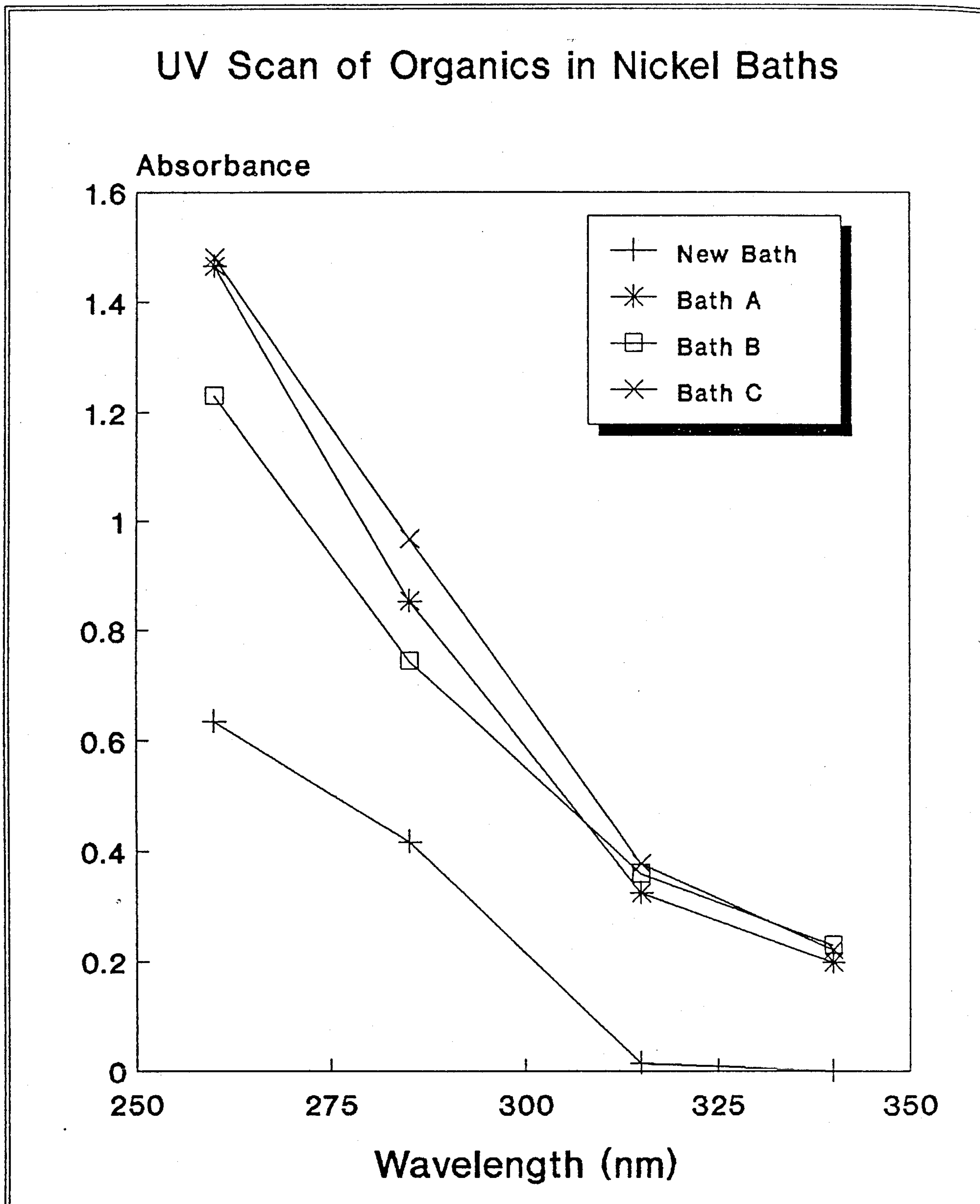


Photomicrographs comparing electrokenetically bonded carbon (EBC) to Fibers (right) to a suspension of powdered activated carbon (left).

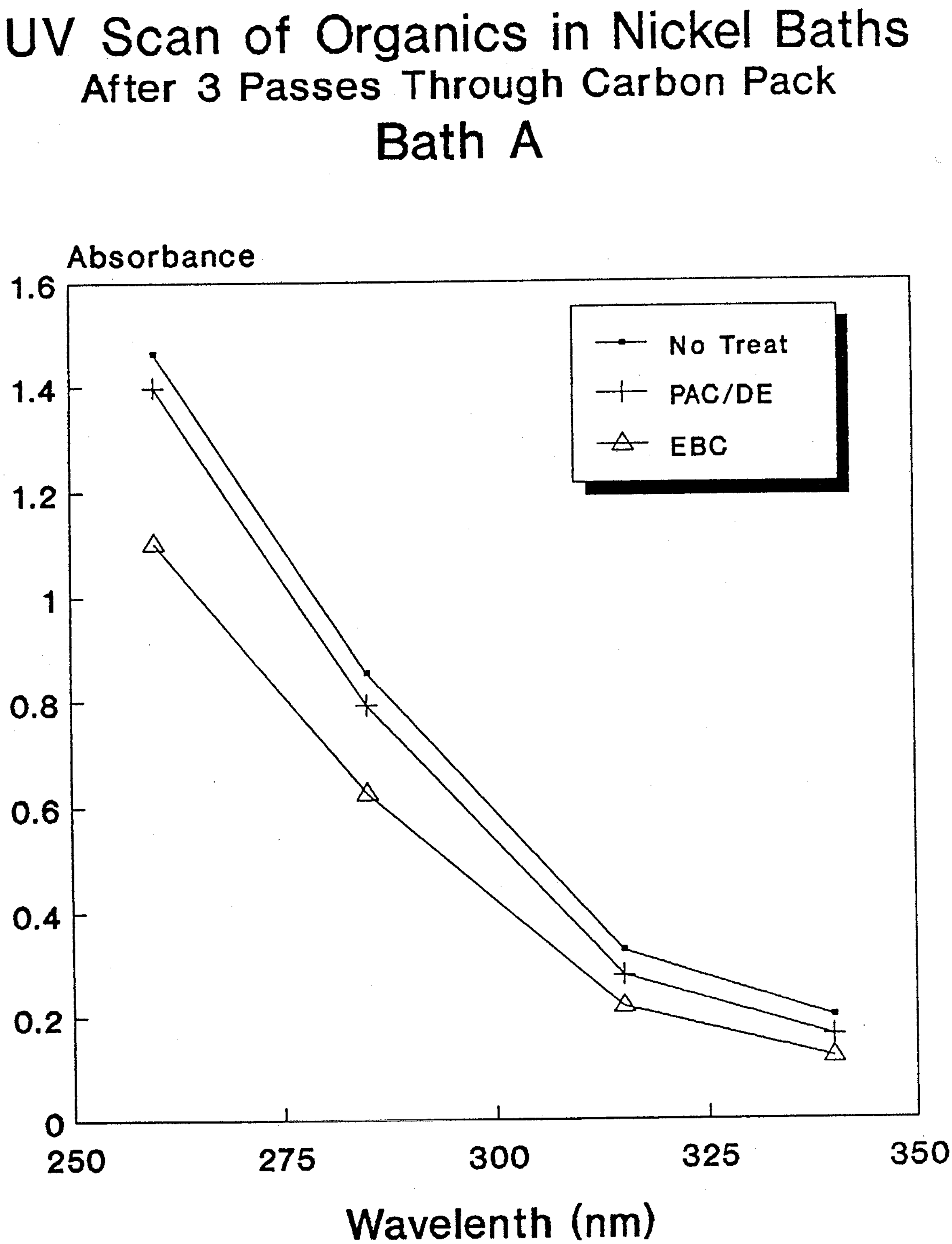


Pressurized Precoat Filtration Apparatus

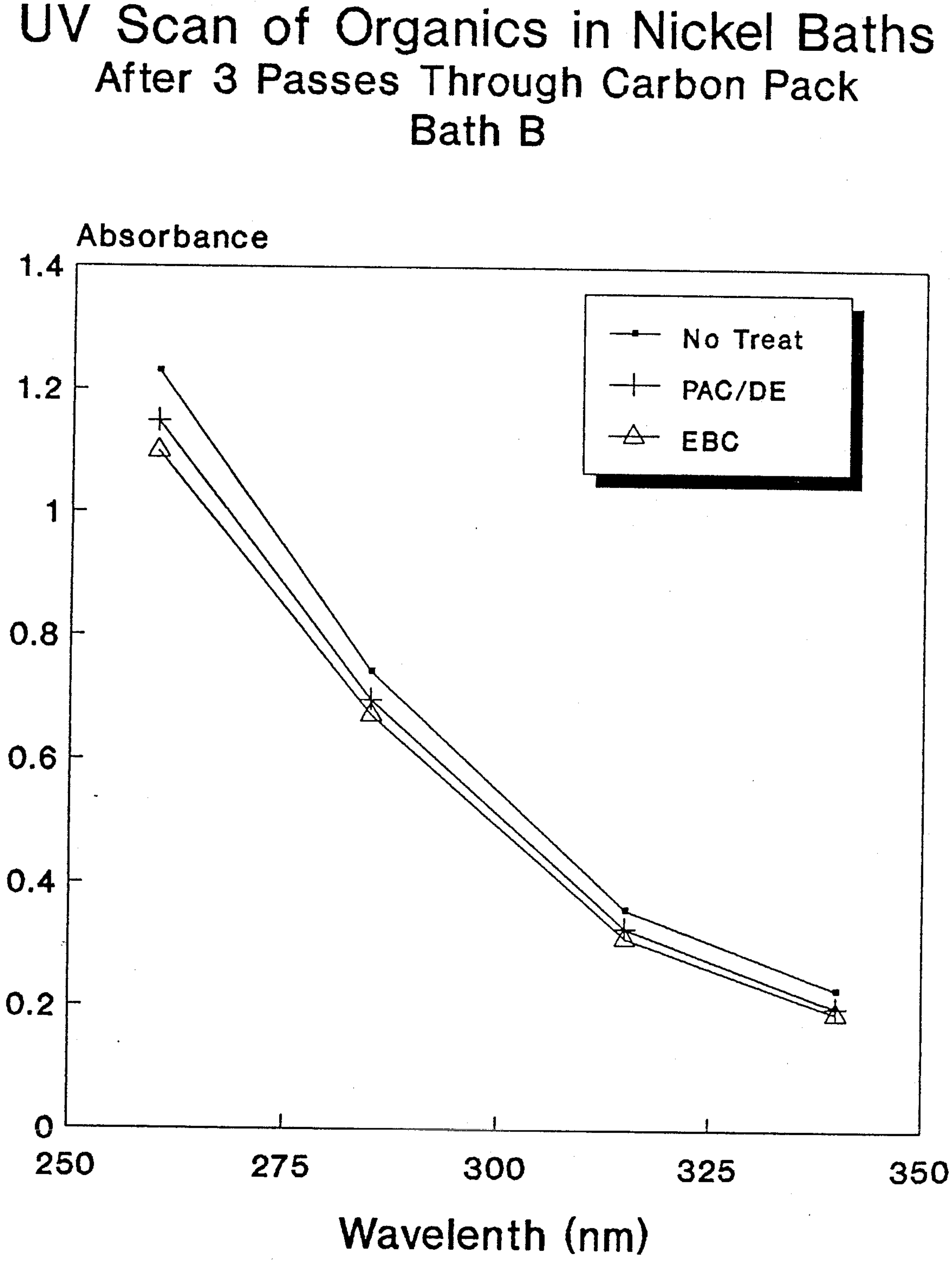
Figure 3


UV Scan of Organics in Nickel Baths
Absorbance
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
New Bath
Bath A
Bath B
Bath C
250
275
300
325
350
Wavelength (nm)

Graph 1

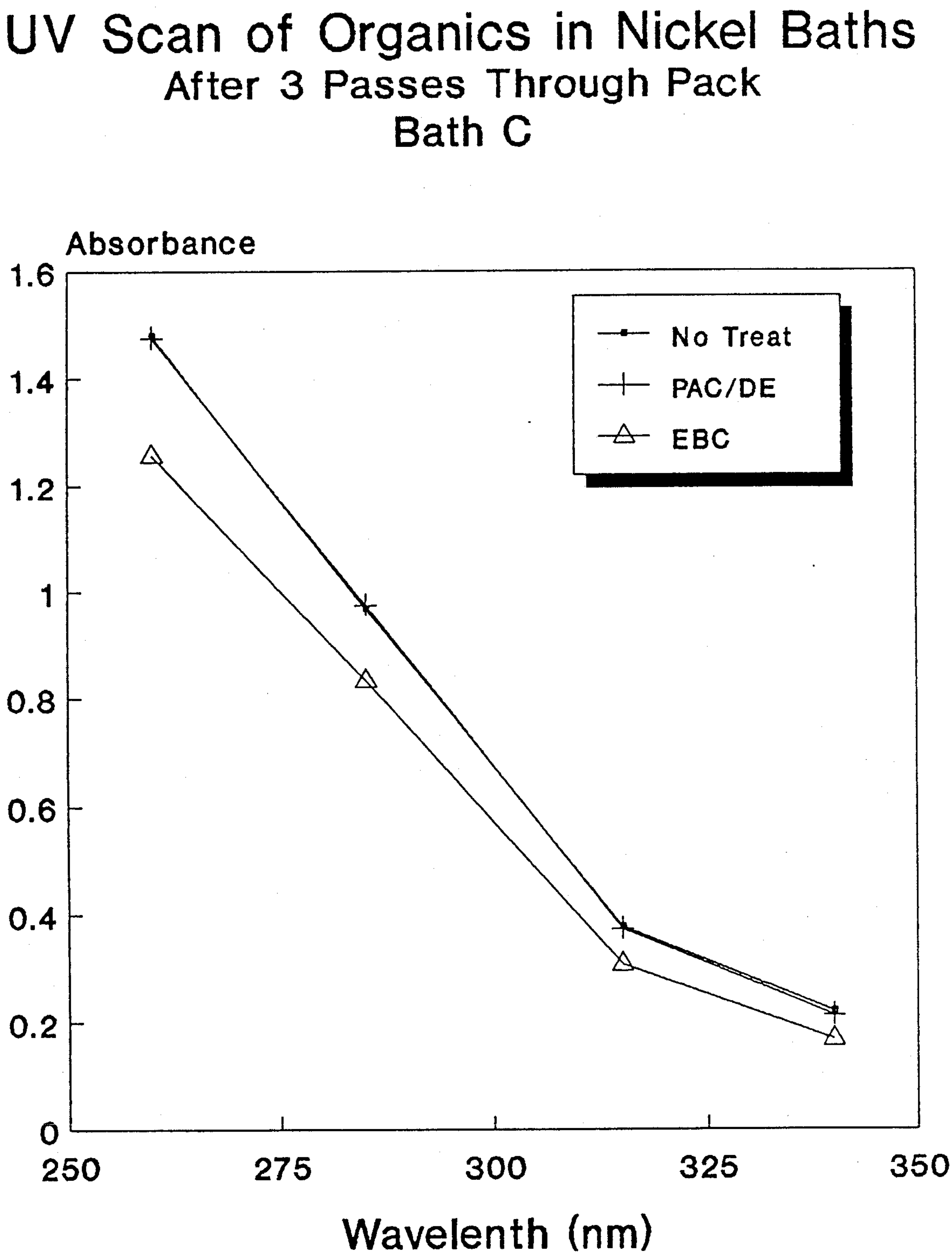

UV Scan of Organics in Nickel Baths
After 3 Passes Through Carbon Pack
Bath A
Absorbance
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
250
275
300
325
350
No Treat
PAC/DE
EBC
Wavelenth (nm)

Graph 2



UV Scan of Organics in Nickel Baths
After 3 Passes Through Carbon Pack
Bath B
Absorbance
1.4
1.2
1
0.8
0.6
0.4
0.2
0
250
275
300
325
350
Wavelenth (nm)
No Treat
PAC/DE
EBC

Graph 3


UV Scan of Organics in Nickel Baths
After 3 Passes Through Pack
Bath C
Absorbance
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
No Treat
PAC/DE
EBC
250
275
300
325
350
Wavelenth (nm)

Graph 4