Electroless Nickel Plating of Polyester Fiber

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ABSTRACT: The optimization of the electroless nickel plating process used for textile applications was investigated with the orthogonal array testing strategy. It was found that the electroless nickel plating process conducted at 40°C and pH 10 for 20 min was the most effective method for improving the metal adhesion performance. The performance of nickel-plated polyester fabrics was confirmed to be dependent on the amount of nickel particles adhering to the fabric surface. The properties of the optimized nickel-plated polyester were enhanced in terms of the fabric weight, fabric thickness, and tensile strength. However, there was a moderate decrease in the tearing strength and crocking fastness. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 108: 2630–2637, 2008

Key words: polyesters; surfaces

INTRODUCTION

Electroless nickel plating is a popular method of combining nonconductive substrates with nickel without the involvement of electricity.[1–5] It is an autocatalytic chemical reduction method that is known as autocatalytic plating. In this plating method, the metallic ions are reduced to metal only on a specific surface in the presence of catalysts before the beginning of the reaction. The autocatalytic nature of the process indicates that the plating is continued as long as the substrate is immersed in the plating bath, giving the unique feature of unlimited nickel-film thickness without the application of a current to the substrate. This technology has been developed for plating nonconductors to provide precious work in manufacturing.[6–8] It is widely used in engineering sectors of different industries; applications include machine frames, base plates, fixtures, and some machine parts with metal-to-metal wear applications.

The metallization of polymer materials by electroless nickel plating has attracted increasing attention over the past few years.[9–12] Electroless nickel plating on textile materials provides functions such as electrical conductivity, antistatic effects, and brilliant decorative effects. Because of its wide range of technological applications, electroless nickel plating has great potential in textile industries.[13–15] In previous research, the possibility of applying the electroless nickel plating process to textile design has been explored.[16,17] To apply this technique to industrial use properly, optimization of the effectiveness of the nickel particle deposition on fabric is required. It is believed that the optimized electroless nickel plating method can provide useful information for industries to obtain greater quality and stable plating. To maximize the amount of nickel particles adhering to the fabric surface, optimization of the deposition condition of electroless nickel plating is investigated in this article. The process parameters, such as the time, temperature, and pH at the deposition stage, are analyzed through a statistical analysis method, and the results are discussed thoroughly in this article.

EXPERIMENTAL

Materials

One hundred percent polyester plain-weave fabric with a white color was supplied by Toyo Menka Kaisha, Ltd. (Japan), and the fabric specifications are summarized in Table I. The fabric samples were cut to the size of 20 cm × 20 cm. Five pieces of fabric were prepared for each trial, and 45 pieces of fabric in all were used for the optimization experiments. All fabric samples were conditioned under standard conditions at a relative humidity of 65 ± 2% and at 21 ± 1°C for at least 24 h before the experiments.

Parameters used for optimizing the electroless nickel plating process

The electroless nickel plating process is practically divided into five main stages: (1) precleaning, (2) sensitization, (3) activation, (4) electroless nickel deposition, and (5) posttreatment. A variety of factors influence the plating performance during the whole

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TABLE I
Fabric Specification

Fabric type		100% polyester
Fabric structure		Plain weave
Fabric weight (g/m^2)		77
Yarn count (tex)	Warp	8.4
	Weft	8.7
Fabric density (sett)	Ends/cm	49
	Picks/cm	38

TABLE II
Parameters and Levels Used in OATS

	Parameter		
Level	Time (min)	Temperature (°C)	pH
I	10	25	8
II	20	40	9
III	30	55	10

electroless nickel plating process. Compared with the sensitization and activation stages, the electroless nickel deposition stage is relatively crucial in affecting the result of metal deposition and is the only factor influencing the metal adhesion efficiency. To achieve the best effect of metal adhesion on polyester fabrics, the optimization of the deposition condition of electroless nickel plating was investigated with an $L_9(3)^4$ orthogonal array testing strategy (OATS) technique. Three variables, that is, time, temperature, and pH, being studied in the deposition process were used for the nickel plating solution composition. Three levels were set for each variable, whereas the decided levels were based on the conventional electroless nickel plating recipe.[17,18]

According to the rules of the OATS technique, nine test runs were generated by the combination of the chosen parameters and the levels of each parameter. The experiments were performed in random order, and each test run was repeated five times to obtain a more accurate result. The details of the selected parameters, levels, and arrangement of experimental trials are summarized in Tables II and III.

The polyester fabrics were plated by means of the electroless nickel deposition process in accordance with the condition requirements stated in Table III, whereas the pretreatment processes, that is, precleaning, sensitization, and activation, and posttreatment process, that is, curing, were kept constant.

In the electroless nickel plating process, all the chemicals were analytical-reagent-grade unless otherwise stated. To facilitate the metal particles plated on the fabric surface, the pretreatment processes, consisting of precleaning, sensitization, and activation, were conducted. During the pretreatment processes, all the fabric samples were precleaned in a 2% nonionic detergent at pH 7 and 40°C for 20 min. Deionized water was then used for rinsing the precleaned samples. In the case of sensitizing fabric surfaces, the cleaned fabric samples were subjected to a surface sensitizer consisting of a mixture of 5 g/L stannous chloride and 5 mL/L hydrochloric acid with slow agitation for 10 min at 25°C and pH 1. The sensitized fabrics were rinsed with deionized water subsequently. The fabric samples were finally immersed in an activator composed of 0.3 g/L palladium(II) chloride, 0.5 mL/L hydrochloric acid (37% concentration), and 20 g/L boric acid at pH 2 and 25°C for 5 min to achieve surface activation. The activated fabrics were finally rinsed with deionized water.

During the deposition stage, the nickel plating solution employed in the electroless nickel plating tank contained 15 g/L nickel (II) sulfate 7-hydrate, 8 g/L trisodium salt dehydrate, 18 g/L ammonium chloride, a few drops of sodium hydroxide (10% concentration), and 15 g/L sodium hypophosphite monohydrate. The activated fabrics were immersed in the nickel plating solution according to the experimental arrangement stated in Table III with constant stirring for the metallizing reaction.

During the posttreatment stage, all the nickel-plated fabrics were rinsed with deionized water at 40°C for 20 min right after the electroless nickel deposition of nickel plating. The cleaned nickel-plated fabrics were then cured with a steaming machine directly at 150°C for 1 min. After electroless nickel plating, the fabric samples were conditioned under standard conditions at a relative humidity of 65 ± 2% and at 21 ± 1°C for at least 24 h before further evaluation.

Conventional electroless nickel plating process

To verify the effectives of the optimized electroless nickel plating process, the conventional electroless nickel plating process was conducted on the basis of the five main stages mentioned previously and similarly to the optimization process, except for the deposition stage. During the deposition stage, the activated fabrics were immersed in the nickel plating so-

TABLE III
Experimental Arrangement

	Parameter		
Test run	Time (min)	Temperature (°C)	pH
1	10	25	8
2	10	40	9
3	10	55	10
4	20	25	9
5	20	40	10
6	20	55	8
7	30	25	10
8	30	40	8
9	30	55	9

lution at pH 9 and 40°C for 20 min with constant stirring for the metallizing reaction.[17,18] After electroless nickel plating, the fabric samples were conditioned under standard conditions at the relative humidity of 65 ± 2% and at 21 ± 1°C for at least 24 h before further evaluation.

Weight change

The fabric weight was measured with a BP211D electronic balance (Sartorius, Germany), and the percentage change of the fabric weight was calculated as follows:

$$\text{Weight change}\,(\%) = \frac{W - W_0}{W_0} \times 100\% \qquad (1)$$

where W is the weight of the substrate after treatment (g) and W_0 is the initial weight of the substrate (g).

Scanning electron microscopy (SEM)

The surface morphology of the test specimens was investigated with a Stereoscan 440 scanning electron microscope (Leica, Cambridge, England).

Fabric thickness

The fabric thickness of the test specimens was measured with a fabric thickness tester (Telex 57767, Hans Baer AG, Zurich, Switzerland) with a pressure of 10 g/cm^2. After the measurement, the result of the change in the fabric thickness was calculated as follows:

$$\text{Change in thickness}\,(\%) = \frac{T - T_0}{T_0} \times 100\% \qquad (2)$$

where T is the fabric thickness of the substrate after treatment (mm) and T_0 is the initial fabric thickness of the substrate (mm).

Tensile strength

The tensile properties of the fabric specimens were evaluated in accordance with ASTM D 5034-95 with a tensile testing machine (Instron 4411, Instron).

Tearing strength

The tearing strength of the fabric specimens was evaluated with an Elmendorf tearing tester (Thwing-Albert Instrument Co.) according to ASTM D 1242-96.

Color fastness to crocking

Color fastness to crocking of the nickel-plated fabric samples in both warp and weft directions under dry and wet conditions was measured in accordance with AATCC 8-2004 (color fastness to crocking).

RESULTS AND DISCUSSION

Optimum conditions for the electroless nickel plating process

Measurement of weight change is the most direct and simplest method to assess the number of nickel particles approaching the fabrics during electroless nickel plating. The more nickel granules are deposited onto the fabric surface, the heavier the fabric weight will be. Therefore, the deposition efficiency can be simply reflected by the change in the fabric weight. The ratio of the weights of the untreated fabric and nickel-plated fabric has been used to express the deposition efficiency, that is, the yield of the electroless nickel deposition. A positive change implies a gain in fabric weight, whereas a negative change indicates a loss in fabric weight. Table IV shows the change in the fabric weight (%) obtained from the nine trials generated by the OATS technique.

Effect of time on the electroless nickel plating process

The effect of the plating time during electroless deposition on the change in fabric weight has been studied. The calculated results, shown in Table IV, indicate that the time factor for electroless nickel plating is a less significant variable in affecting deposition efficiency than the factors of temperature and pH. The deposition efficiency, reflected by the change in fabric weight against the deposition time during the electroless nickel plating process, is presented in Figure 1.

The fabric weight increases noticeably in the first 10 min, and this indicates that there are a lot of nickel particles adhering to the untreated polyester fabrics. However, the growth of fabric weight between 10 and 20 min of electroless plating increases slightly from 32.20 to 32.40%. This shows that there is an increase in the difficulty of depositing nickel particles onto the fabric surface after the deposition of nickel granules onto the fabric surface in the first 10 min. Furthermore, the curve exhibits a declining tendency when the deposition process exceeds 20 min. This demonstrates that the saturation effect does occur in the nickel deposition solution.

When the electroless plating process reaches its peak at 20 min, the penetrated nickel particles are

TABLE IV
Orthogonal Table for Optimization of Nickel Plating

Test run	Parameter			Change in fabric weight (%)
	Time (min)	Temperature (°C)	pH	
1	10	25	8	0.22
2	10	40	9	14.97
3	10	55	10	17.01
4	20	25	9	0.33
5	20	40	10	18.13
6	20	55	8	13.95
7	30	25	10	0.33
8	30	40	8	13.85
9	30	55	9	15.45

$\sum$ Change in fabric weight (%)	Parameter		
	Time	Temperature	pH
$\sum$ I	32.20%	0.88%	28.02%
$\sum$ II	**32.41%**	**46.95%**	30.75%
$\sum$ III	29.63%	46.41%	**35.47%**
Difference	*2.78%*	*46.07%*	*4.72%*

Bold numbers are the greatest values among all the values shown at the levels of the different factors used, whereas italic numbers show the level of importance of each factor.

released from fabrics to solution again because of a saturation effect leading to a subsequent loss in fabric weight. The longer the time is for the polyester fabrics to be immersed in the deposition solution, the more the nickel particles will diffuse back to the deposition solution bath when the fabric is saturated. Figure 1 shows that the nickel adhesion cannot be improved further by prolongation of the deposition time after its saturation, and the optimized time for nickel deposition is confirmed to be 20 min.

Effect of temperature on the electroless nickel plating process

The effect of the plating temperature on the change in fabric weight during electroless nickel plating has also been examined. With reference to the calculated results shown in Table IV, the amount of energy or heat present in the electroless nickel plating solution is confirmed to be the most important variable affecting the effectiveness of deposition in comparison with the other two factors, that is, time and pH. Figure 2 presents the deposition efficiency, in terms of fabric weight change, against the temperature during the electroless nickel plating process.

The temperature of the electroless nickel deposition solution has a strong effect on the process performance, as reflected by more than 45% growth in fabric weight after deposition. Figure 2 clearly shows that the fabric weight has less than 1% growth when the deposition process is conducted at 25°C.

The considerable increase in fabric weight corresponds to the rise of the deposition temperature from 25 to 40°C. When the deposition temperature is above 40°C, the fabric weight shows only a slight decrease. However, it can still maintain growth of fabric weight at about 45% in comparison with the fabric deposited at 25°C.

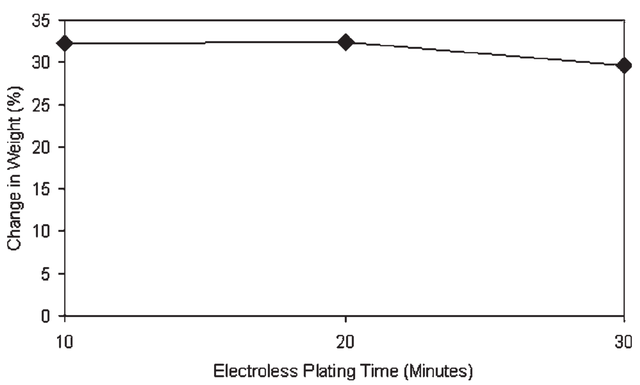


Figure 1 Effect of different electroless plating times on the weight change.

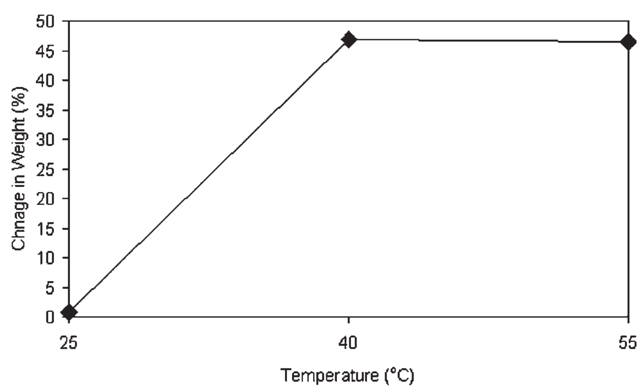


Figure 2 Effect of different electroless plating temperatures on the weight change.

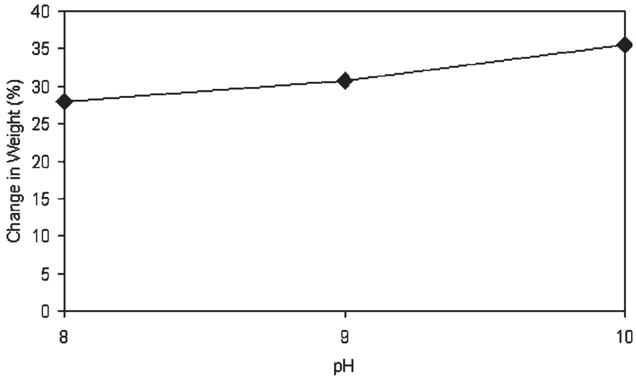
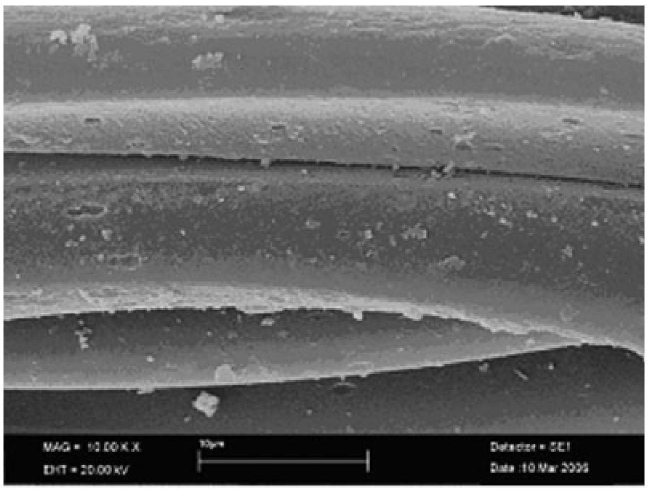
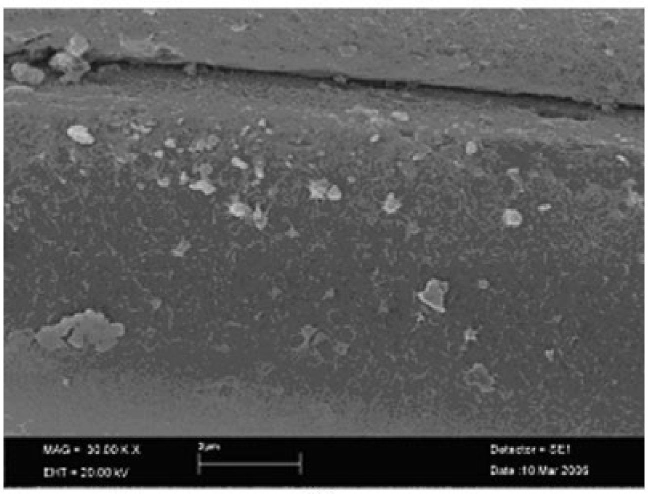


Figure 3 Effect on different pHs during the electroless plating process on the weight change.

Figure 2 also shows that the change in the plating temperature can greatly affect the deposition efficiency. To a certain extent, the performance of nickel

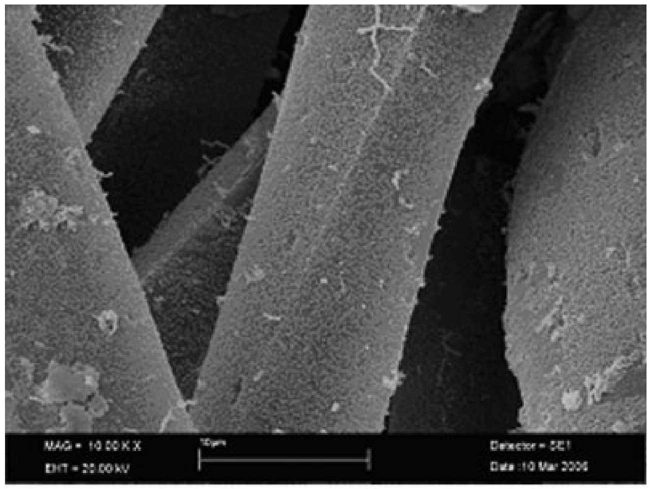


(a)

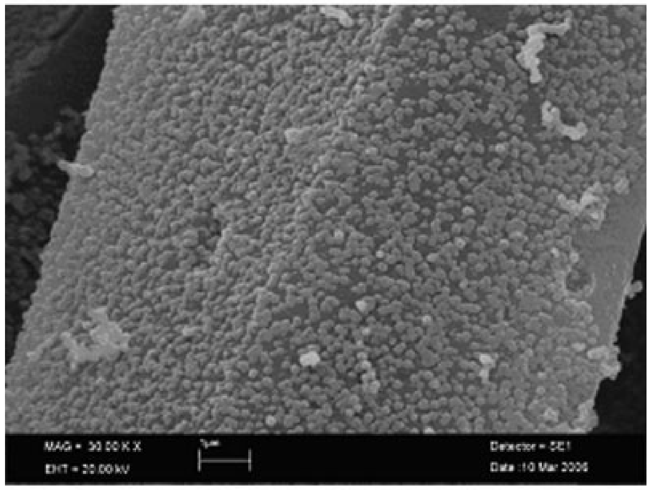


(b)

Figure 4 Scanning electron micrographs of conventional electroless nickel-plated polyester: (a) 10,000× and (b) 30,000×.



(a)



(b)

Figure 5 Scanning electron micrographs of optimized nickel-plated polyester: (a) 10,000× and (b) 30,000×.

deposition can be improved further by the plating temperature of the deposition stage being raised. Moreover, operation below the recommended range yields ineffective deposition results as well as a slow plating rate. It can also cause poor adhesion and plating because of improper initiation. On the basis of the experimental results, the optimized temperature determined for nickel deposition is confirmed to be 40°C.

Effect of pH on the electroless nickel plating process

The effect of the pH of the electroless nickel plating solution on the change in fabric weight during the deposition process has been investigated as well. Table IV reveals that the pH used for the plating solution to affect the nickel adhesion reaction is a variable that is less significant than temperature but more significant than time. The pH of the nickel

TABLE V
Fabric Weight of Nickel-Plated Polyester Fabrics

Sample	Conventional electroless nickel-plated polyester fabrics	Optimized electroless nickel-plated polyester fabrics
Original weight (g)	3.08	3.08
Weight after electroless nickel plating process (g)	3.38 (↑9.87%)	3.64 (↑18.15%)

plating solution is adjusted by a sodium hydroxide solution, and the influence of pH on the yield of nickel deposition on fabrics is shown in Figure 3.

The results in Figure 3 indicate that the nickel deposition performance is sensitive to pH with a positive relationship; that is, the fabric weight exhibits an increasing tendency. When the pH of the plating solution is increased, the weight of nickel-plated fabrics increases correspondingly. The fabric weight increases from 28% at pH 8 to 35% at pH 10; this is almost 7% growth in weight. It can be concluded that pH can serve the function of increasing the fabric weight in the electroless nickel plating process.

The slope of the curve with respect to the change in fabric weight between pHs 9 and 10 is steeper than that between pHs 8 and 9. This implies that the higher the pH value is, the more nickel particles will be deposited onto the fabric surface. Figure 3 shows that the performance of nickel adhesion may be enhanced further by an increase in the pH of the electroless nickel solution beyond 10 during the deposition process. To minimize the polyester fabric damage under an alkaline condition, that is, pH beyond 10, the optimized pH selected for nickel deposition is 10 in this study.

On the whole, the optimum condition of electroless nickel plating has been successfully developed. The newly developed optimum condition of electroless nickel plating is able to enhance the achievement of maximizing the coating of metal particles on a fabric surface, as reflected by the increase in fabric weight. It is concluded that all three factors used, including the time, temperature, and pH, can affect the efficiency of electroless nickel deposition on polyester fabrics. Of the three factors being studied, temperature is the most dominant factor affecting the deposition efficiency, followed by pH and time. By the calculation of the results obtained from the nine trials, the newly developed optimum condition acquired for the electroless nickel deposition is 40°C, and the deposition time is 20 min, with the pH of the electroless nickel deposition solution being maintained at pH 10.

SEM images

SEM images were captured for studying the change in the surface morphology of the optimized nickel-plated polyester fabrics. The surfaces of (1) nickel-plated polyester fibers with conventional deposition conditions and (2) nickel-plated polyester fibers with optimum deposition conditions are presented in Figures 4 and 5, respectively, with a magnifications of 10,000 and 30,000×.

Both figures clearly illustrate that the surfaces of nickel-plated polyester fibers are covered by nickel particles. The nickel particles are unevenly distributed on the fiber surfaces treated with conventional nickel deposition. On the contrary, nickel particles deposited onto the optimized nickel-plated fibers are more uniformly distributed throughout the fiber. Furthermore, Figure 5 obviously shows that the amount of nickel granules deposited onto the nickel-plated fibers treated with the optimized electroless nickel deposition condition is drastically increased and denser in comparison with that of the conventional electroless nickel-plated polyester fibers, as shown in Figure 4. Another important point is that the sizes of the nickel particles deposited onto the optimized nickel-plated fibers are almost the same. As for the conventional electroless nickel-plated polyester fibers, the sizes of the nickel particles are varied with different dimensions. The overall results evidence that the optimized electroless nickel plating is sufficient and effective for improving the performance of nickel adhesion on the polyester fabrics.

TABLE VI
Fabric Thickness of Nickel-Plated Polyester Fabrics

Sample	Conventional electroless nickel-plated polyester fabrics	Optimized electroless nickel-plated polyester fabrics
Original thickness (mm)	0.270	0.270
Thickness after electroless nickel plating process (mm)	0.272 (↑0.74%)	0.277 (↑2.59%)

TABLE VII
Tensile Strength in Warp and Weft Directions of Nickel-Plated Polyester Fabrics

Sample	Conventional electroless nickel-plated polyester fabrics	Optimized electroless nickel-plated polyester fabrics
Breaking load in warp direction (N)	309.2	359.4 (↑16.24%)
Breaking load in weft direction (N)	250.9	259.0 (↑3.22%)
Average breaking load (N)	280.1	309.2 (↑10.40%)

Weight change

The calculated changes in fabric weight of (1) nickel-plated polyester fabrics with conventional electroless nickel plating and (2) nickel-plated polyester fabrics with optimized electroless nickel plating are presented in Table V.

With reference to Table V, both the conventional electroless nickel plating and optimized electroless nickel plating contribute to the increase in fabric weight. However, the fabric weight in the latter condition is heavier than that of the former one. On the basis of the results shown in Table V, it is concluded that the percentage growth of the fabric weight of polyester fabrics treated under the optimum electroless nickel plating condition is almost twice that of polyester fabrics treated under the conventional electroless nickel plating condition. The fabric weight of the former condition is 3.64 g, whereas that of the latter condition is only 3.38 g. Table V shows that the optimized nickel plating can provide an efficient metal adhesion reaction on the polyester fabrics, resulting in 18% growth in weight.

Fabric thickness

The changes in fabric thickness of nickel-plated polyester fabrics under conventional electroless nickel plating and optimized electroless nickel plating are summarized in Table VI.

There is an increase in the thickness of the fabric surface, as illustrated in Table VI. It is probably due to the coating of a thin layer of metal on the fabric surface. Fabric thickness generally increases after an electroless nickel plating treatment. The fabrics treated with optimized electroless nickel plating are thicker than those treated with conventional electroless nickel plating. The thickness of polyester fabrics under the conventional deposition condition increases from 0.270 to 0.272 mm, whereas the thickness of polyester fabrics under the optimum condition increases from 0.270 to 0.277 mm. Table VI proves that the newly developed optimum condition does provide an effective adhesion reaction on the polyester fabrics and contributes almost a 3% improvement of the fabric thickness.

Tensile strength

The results of the mean tensile strength in both warp and weft directions and the mean tensile strength for each treatment are shown in Table VII.

Table VII demonstrates that the tensile strength of nickel-plated polyester fabrics under the optimized electroless nickel plating condition is higher than that under the conventional electroless nickel plating condition. Table VII also shows that the tensile strength increases in both warp and weft directions. Because there is a layer of metal deposited onto the fabric surface, this can create extra strength on yarns, resulting in the enhancement of the tensile strength. The average maximum breaking load grows from 280 to 309 N.

On the basis of the information shown in Table VII, the growth of the tensile strength in the warp direction is greater, that is, 5 times more than that in the weft direction. As a result, the maximum strength required to break the fabrics grows significantly in the warp direction, most likely because the warp yarns have tighter construction than the weft yarns. This will lead to larger surface contact of warp yarns with those nickel particles present in the plating solution during electroless nickel deposition.

Tearing strength

Table VIII shows the results of the mean tearing strength in both the warp and weft directions and the average tearing strength.

TABLE VIII
Tearing Strength in Warp and Weft Directions of Nickel-Plated Polyester Fabrics

Sample	Conventional electroless nickel-plated polyester fabrics	Optimized electroless nickel-plated polyester fabrics
Tearing forces in warp direction (N)	8.47	7.37 (↓12.96%)
Tearing forces in weft direction (N)	7.53	6.90 (↓8.33%)
Average tearing force (N)	8.00	7.14 (↓10.78%)

TABLE IX
Color Fastness to Crocking

Treatment		Conventional electroless nickel-plated polyester fabrics	Optimized electroless nickel-plated polyester fabrics
Dry	Warp	Grade 3	Grade 2–3
	Weft	Grade 2–3	Grade 2–3
Wet	Warp	Grade 3	Grade 2–3
	Weft	Grade 2–3	Grade 2–3

Table VIII indicates that the tearing strength of nickel-plated polyester fabrics treated under the conventional electroless nickel plating condition is stronger than that of nickel-plated polyester fabrics treated under the optimized electroless nickel plating condition. The nickel-plated polyester fabrics become more brittle after the plating process under the optimized electroless nickel plating condition in comparison with those treated under the conventional electroless nickel plating condition. Table VIII also reveals that the tearing strength decreases in both the warp and weft directions. This may be due to the fact that a higher concentration alkaline solution with a high pH attacks fabrics and degrades their mechanical properties. The reduction in the tearing strength of warp yarns is more significant than that of weft yarns, that is, almost a 5% drop. This is probably due to the fact that more warp yarns are exposed to the deposition solution than weft yarns as a result of tighter fabric construction.

Color fastness to crocking

The results of color fastness to crocking were assessed by two observers according to the AATCC specification and reported on a grade scale, as shown in Table IX.

Table IX shows that under the optimized deposition condition, the staining results of nickel-plated polyester fabrics have a decreasing tendency. In the case of the conventional nickel plating condition, the staining results are rated as grade 3 and grade 2–3 for the warp and weft directions, respectively, when conducted under both dry and wet rubbing conditions. However, under the optimized deposition condition, all the staining results are rated as grade 2–3 for both the warp and weft directions when conducted under dry and wet rubbing conditions. This implies that more nickel granules are lost from the fabric surface during rubbing action after subjection to the optimized nickel plating process. This is probably due to the fact that more nickel particles deposit onto the fabric surface under the optimized condition, as reflected by the increment of the fabric weight and thickness. The more nickel particles are deposited onto the fabric surface, the more severe the color stains after crocking will be.

CONCLUSIONS

In this study, the optimization of electroless nickel plating was investigated with OATS. It was found that the electroless nickel plating process at the deposition stage with a temperature of 40°C, a time of 20 min, and a pH of 10 was the most effective method in improving the metal adhesion performance. Trials were performed to examine and compare the effects of optimized electroless nickel plating on the properties of nickel-plated polyester fabrics. The performance of nickel-plated polyester fabrics was confirmed to be dependent on the number of nickel particles adhering to the fabric surface. The properties of optimized nickel-plated polyester were enhanced with respect to SEM analysis, fabric weight, fabric thickness, and tensile strength, but there was a moderate decrease in the tearing strength and crocking fastness.

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