

**GREENER METHOD OF COLORATION ON CONDUCTIVE TEXTILE SUBSTRATES****Kalapriya, K. and *Gurumallesh Prabu, H**

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ARTICLE INFO**Article History:**Received 15th July, 2012Received in revised form 25th, July, 2012Accepted 20th August, 2012Published online 30th August, 2012**Key words:**Conducting polymer, Cotton, Dyeing,
Polyester, Silk, Surfactant**ABSTRACT**

Dyeing on textile substrates such as cotton, silk and polyester was carried out by the conventional method. Dyeing was carried out in open dye bath. Cotton fabrics were dyed with Reactive, Direct and Vat dyes. Silk fabric was dyed with Acid dyes. Polyester fabric was dyed with Disperse dyes. In another method and in order to produce color on conductive fabric by a greener route (without using auxiliaries and heat), fresh textile substrates were subjected to electrochemical polymerization with pyrrole and then immersed in the dye solution at room temperature. The effects of surfactants (sodium dodecyl sulphate, Cetyl trimethylammonium bromide) and dopants (n-tetrabutylammonium tetrafluoroborate, 1-butyl-3-methylimidazolium tetrafluoroborate) in the polymerization were studied. The polymerized fabric containing the dye (colored) was then measured using Computer Color Matching (CCM) analysis to assess the dye uptake. Crease recovery angle of the fabrics was measured. Electrical conductivity of conductive fabrics was measured by electrochemical impedance spectroscopy. Surface morphology was tested with SEM images.

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INTRODUCTION

Conductive polymers have attracted the attention of a great number of researches in the textile field due to their potential applications in composites with natural, artificial or synthetic fibers (Malinauskas, 2001; Kim *et al.*, 2003; Dhawan *et al.*, 2002; Hakansson *et al.*, 2004; Hu *et al.*, 2005; Kathirgamanathan *et al.*, 2000). Electro conductive textiles approach supports the idea of a smooth combination of the two worlds of electronics and clothing. Trends in electro-conductive textiles and smart fabrics try to find a symbiosis of electronics and clothing (Mythili *et al.*, 2007). The emerging areas of conducting materials, electronic and interactive textiles have been explored with the recent innovations in smart textiles (Jain *et al.*, 2005). Fabrics coated with or containing conductive polymers are suitable for technological applications such as electromagnetic interference (EMI) shielding (Kim *et al.*, 2003; Dhawan *et al.*, 2002), heat generation (Hakansson *et al.*, 2004), cooling system (Hu *et al.*, 2005) and electro-static discharge (Kathirgamanathan *et al.*, 2000). Researchers have indicated that modification by conductive polymers seems to be the interesting approach enabling nature fibres, new conductive functionality (Micusik *et al.*, 2006; Hosseini *et al.*, 2005) because of convenient polymerization and good conductivity. Polypyrrole is one of the most suitable conductive polymers for deposition on textile materials due to its excellent conductivity and relevant environmental stability (Kim *et al.*, 2002; Varesano *et al.*, 2005). Electrochemical polymerization of pyrrole is traditionally performed in an electrolyte / molecular solvent system. The processing parameters, particularly the size and nature of the dopant counter-ions from the solvent / electrolyte system, and the nature of the solvent itself, can have a marked influence on the properties of the resultant polymer film

Wallace *et al.*, 2003). Conductive fibers are used for many thermal applications such as flexible heating pads, electrical heating blankets, and jackets (Ghosh *et al.*, 2005). Nanotechnology is being applied to produce conducting textiles to eliminate multiple antennas used for data and power transmission in soldier clothing (Jain *et al.*, 2005). Electrotiles may find applications in other markets, such as healthcare, safety, communication, entertainment and protective clothing (Karthik, 2004).

Electrochemically and chemically coated cotton fabrics with polypyrrole were comparatively evaluated and characterized in order to produce the conducting fabrics (Firoz Babu *et al.*, 2009). Cotton, silk, wool and nylon have been coated using chemical and electrochemical polymerization in the presence of the host fibre with no significant difference or an increase in the breaking load for the treated fibres (Nouri *et al.*, 2000; Kaynak *et al.*, 2002). Electrical conductivity of silk, cotton, polyester and wool fibres electrochemically coated with polypyrrole was in the range between 0.2 and 16 Scm⁻¹. The conductivity of wool and polyester yarns coated with polyaniline were considerably lesser than cotton, acrylic and nylon (Nouri *et al.*, 2000). Potentiodynamic and potentiostatic synthesis of polyaniline on conducting fabrics of PES-Ppy/AQSA have been achieved. Electrochemical impedance measurements confirmed that Pani films electrosynthesized on PES/Ppy-AQSA improved the electronic conductivity of the samples (Molina *et al.*, 2009). Hakansson *et al.*, 2006 studied the effects of the dopants on density, optical absorption and conductivity of the polypyrrole.

The objective of this study was to experiment and discuss the fixation (binding) of dye on cotton, silk and polyester fabrics by (i) conventional dyeing method and (ii)

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electrochemical polymerization of pyrrole in the presence of dye method. The second method was performed by greener concept with an objective to avoid the use of dye auxiliaries and heat to impart coloration in polymerized (conductive) substrates.

MATERIALS AND METHODS

Desized, scoured and bleached cotton fabric, dil.HCl treated silk and polyester fabrics were used in this study. All chemicals used were of AR grade from Merck. In the first conventional method, dyeing on textile substrates such as cotton, silk and synthetic polyester was carried. For this, cotton fabric was dyed with Reactive dyes such as Reactive Blue 4 (RB 4), Reactive Orange 4 (RO 4), Reactive Violet 5 (RV 5) and Direct dyes such as Direct Red 7 (DR 7), Direct Black 22 (DB 22), Direct Blue 1 (DB 1) and Vat dyes such as Vat Yellow 2 (VY 2), Vat Red 10 (VR 10), Vat Brown 1 (VB 1). Silk fabric was dyed with Acid dyes such as Acid Yellow 17 (AY 17), Acid Orange 7 (AO 7), Acid Red 73 (AR 73). Polyester fabric was dyed with Disperse dyes such as Disperse Orange 3 (DIO 3), Disperse Orange 13 (DIO 13) and Disperse Red 11 (DIR 11). The stock solutions of dyes were prepared at 1% concentration. In the second modified method to produce color on conductive fabric by a greener route (without using dye auxiliaries and heat), fresh textile substrates were subjected to electrochemical polymerization using an Aplab DC power supply L3230 unit. Platinum coated Titanium mesh as anode and stainless steel as cathode were used. The fabric was wounded with glue over anode as reported by Subianto *et al.*, 2007. The experiment was carried out galvanostatically at 0.1A at room temperature and then immersed in the dye solution. The effects of surfactants (sodium dodecyl sulphate, Cetyl trimethylammonium bromide) and dopants (n-tetrabutylammonium tetrafluoroborate, 1-butyl-3-methylimidazolium tetrafluoroborate) in the polymerization were studied. The polymerized fabric containing the dye was then measured using Gretagmacbeth 2180 UV Computer Color Matching instrument to assess the coloration efficiency. Crease recovery angle of the fabrics was measured. Electrical conductivity of conductive fabrics was measured by using AUTOLAB electrochemical impedance bridge in the frequency range of 50 mHz to 100 KHz at an amplitude of 20 mV, by sandwiching the polymer coated conductive fabric (1 cm dia) between two stainless steel (SS304) electrodes contacts. Surface morphology was tested with SEM images using Hitachi S3000H instrument.

Conventional method of dyeing was carried out in an open dye bath at a material to liquor ratio (MLR) of 1:30. In the dyeing of cotton, the fabric was entered into the dyebath containing NaOH and hydrose. 2% NaCl was added and dyeing was continued for 45 minutes at a temperature of 90°C. Then the dyed fabric was taken out and washed well. In the dyeing of silk, the fabric was entered into the dyebath containing 0.2% acetic acid. After 10 minutes, dye was added. 2% NaCl was added and dyeing was continued for 45 minutes at 80°C. Then the dyed fabric was taken out and washed well. In the dyeing of polyester, the fabric was entered into the dyebath containing ammonium acetate and acetic acid. Dyeing was continued for 45 minutes at 130°C. Then the dyed fabric was taken out and washed well.

In the proposed second method, electrochemical method of polymerization of pyrrole was performed using DC power supply unit. Pyrrole was dissolved in acetonitrile and suitable dilutions were made. The surfactants sodium dodecyl sulphate (SDS) and cetyltrimethyl ammonium bromide (CTAB) were used in the study. Dopants such as n-tetrabutylammonium tetrafluoroborate (TBATFB) and 1-butyl-3-methylimidazolium tetrafluoroborate (BMimTFB) were used to study the effectiveness of polymerization. Variables such as dye, dopant and surfactant were varied. A material to liquor ratio of 1:30 was maintained. Required amount of dye was added to produce 0.5% shade. The fabric was subjected to stirring in the bath containing monomer, surfactant and dopant for 1 h. The polymerized fabric was taken out and immersed in the dye solution for 1 h at room temperature. Polymerized fabric containing the dye was then taken out, washed with water, dried and subjected to dye uptake and other tests.

RESULTS AND DISCUSSION

Under the specified conditions employed in the conventional dyeing of cotton, DB 22 has resulted better dyeability with K/S value of 5.23 (Table 1) and its wash fastness tested gave a K/S of 4.50 (Table 2). Thus, the dyeability and dye fixation were good. The minimum K/S value of 0.64 was obtained with VB 1 dye. In the dyeing of silk, the maximum K/S value of 9.38 was obtained with Acid Yellow 17 dye. The wash fastness was also good. In the dyeing of polyester, the dyeability was moderate in the fabric dyed with DIO 13 and the K/S value of 1.79 was obtained.

Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 5×10^{-2} M SDS was employed. Required amount of dye was added to produce 0.5% shade. With cotton, the K/S value showed its maximum at 2.37 with DB 1 dye and the wash fastness result was acceptable. DB 1 dye has resulted the highest chroma value of 16.38 (Table 3). With silk, the maximum K/S value of 13.18 was obtained with AO 7 dye and the wash fastness was good. A chroma value of 17.45 was obtained with this AO 7 dye. The wash fastness result was very good. With polyester, the maximum K/S value of 2.13 was obtained with DIO 13 dye and the wash fastness was good. The highest chroma value of 25.32 was obtained with DIO 13 dye.

Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 3×10^{-2} M SDS, 5×10^{-2} M TBATFB was employed. For cotton, the maximum K/S value of 7.59 was obtained with DB 1 dye. The wash fastness test showed dye fixation. The highest chroma value of 35.22 was obtained with DB 1 dye. With silk, the maximum K/S value of 12.64 was obtained with AO 7 dye and the wash fastness was noticed as good. The highest chroma value of 58.29 was obtained with AO 7 dye. For polyester, the maximum K/S value of 2.05 was obtained with DIO 13 dye and the wash fastness was observed as moderate. The highest chroma value of 20.08 was obtained with DIO 13 dye.

Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 3×10^{-2} M SDS, 5×10^{-2} M BMimTFB was employed. Maximum K/S value of 4.51 was obtained with DB 1 dye and the wash fastness observed as

Table 1 Comparison of color strength (K/S) for dyed fabric in different coloration systems

Dye	Color strength (K/S) for dyed fabric						
	Conventional	Py /SDS /dye	Py /SDS /TBATFB /dye	Py /SDS /BMimTFB /dye	Py /CTAB /dye	Py /CTAB /TBATFB /dye	Py /CTAB /BMimTFB /dye
Reactive Blue 4	0.77	0.97	0.87	0.86	1.21	1.58	0.97
Reactive Orange 4	0.83	0.99	0.92	0.96	2.02	3.03	1.04
Reactive Violet 5	0.65	1.14	0.66	0.80	1.25	2.95	0.86
Direct Red 7	5.21	2.09	5.89	4.33	4.41	5.71	5.24
Direct Black 22	5.23	1.10	1.16	1.86	4.01	5.54	4.37
Direct Blue 1	4.07	2.37	7.59	4.51	5.02	5.81	4.83
Vat Yellow 2	0.65	0.78	0.80	0.82	1.21	1.39	1.15
Vat Red 10	0.67	0.90	1.00	0.92	1.19	1.71	1.07
Vat Brown 1	0.64	0.84	0.99	1.00	1.21	1.45	1.02
Acid Yellow 17	9.38	10.31	8.93	8.55	9.65	9.35	9.82
Acid Orange 7	8.15	13.18	12.64	8.32	12.89	15.96	10.87
Acid Red 73	8.37	10.54	9.57	12.01	9.23	10.36	10.54
Disperse Orange 3	1.66	1.71	1.92	1.77	1.75	3.09	2.05
Disperse Orange 13	1.79	2.13	2.05	1.54	1.63	1.08	1.99
Disperse Red 11	1.67	1.43	1.84	1.81	1.34	1.85	2.76

good for cotton. The highest chroma value of 24.68 was also obtained with DB 1 dye. Maximum K/S value of 12.01 was obtained with AR 73 dye and the wash fastness was noticed as good for silk. The highest chroma value of 54.87 was obtained with the same AR 73 dye. With polyester, the maximum K/S value of 1.81 was obtained with DIR 11 dye and the wash fastness tested sample showed moderate results. The highest chroma value of 20.90 was obtained with DIR 11 dye

Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 5×10^{-2} M CTAB was employed. With cotton, the maximum K/S value of 5.02 was obtained with DB 1 dye and the wash fastness was good. The highest chroma value of 22.61 was obtained with the same DB 1 dye. With silk, the maximum K/S value of 12.89 was obtained with AO 7 dye and the wash fastness was very good. The highest chroma value of 35.65 was obtained with AO 7. With polyester, the maximum K/S value of 1.75 was obtained with DIO 3 dye and the wash fastness was moderate. The highest chroma value of 15.21 was obtained with DIO 3 dye.

Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 3×10^{-2} M CTAB, 5×10^{-2} M TBATFB was employed. With cotton, the maximum K/S value of 5.81 was obtained with DB 1 dye and the wash fastness was good. The highest chroma value of 33.33 was obtained with DB 1 dye. With silk, the K/S value obtained its maximum at 15.96 with AO 7 dye and the wash fastness was very good. The highest chroma value of 64.29 was obtained with AO 7 dye. With polyester, the maximum K/S value of

3.09 was obtained with DIO 3 dye and the wash fastness was moderate. The highest chroma value of 14.39 was obtained in DIO 3 dye. Polymerization of textile substrates with reactants in the bath containing 5×10^{-2} M Py, 3×10^{-2} M CTAB, 5×10^{-2} M BMimTFB, was employed. With cotton, the maximum K/S value of 5.24 was obtained with DR 7 dye and the wash fastness was good. The highest chroma value of 29.92 was obtained with DR 7 dye. With silk, the maximum K/S value of 10.87 was obtained with AO 7 dye and the wash fastness was very good. The highest chroma value of 46.59 was obtained with AO 7 dye. With polyester, the maximum K/S value of 2.76 was obtained with DIR 11 dye and the wash fastness was good. The highest chroma value of 12.31 was obtained with DIR 11 dye.

“L”, “a” and “b” data represent the lightness or darkness, redness or greenness and yellowness or blueness respectively. It is inferred that higher the L value, lower the dyeability and lower the L value, higher the dyeability. “H” is a measure of hue and is represented as an angle ranging from 0 to 360°. LabCH results were obtained for the samples. If “a” and “b” are both positive, then the hue angle should be between 0° and 90°. It was found that both a and b were positive for AR 73 dye and AO 7 dye. In all these dyed samples, the hue angle falls between 18.06 and 56.70 for dyed fabric. If “a” is negative and “b” is positive, then the hue angle should be between 90° and 180°. It is found that with AY 17 dye, a is negative and b is positive and the hue angle was 100.60 for dyed fabric (Table 4).

Table 2 Comparison of color strength (K/S) for wash fast fabric in different coloration systems

Dye	Color strength (K/S) for wash fast fabric						
	Conventional	Py / SDS / dye	Py / SDS / TBATFB / dye	Py / SDS / BMimTFB / dye	Py / CTAB / dye	Py / CTAB / TBATFB / dye	Py / CTAB / BMimTFB / dye
Reactive Blue 4	0.71	0.79	0.79	0.74	1.09	1.40	0.79
Reactive Orange 4	0.65	0.80	0.80	0.85	1.86	1.81	0.82
Reactive Violet 5	0.60	0.99	0.53	0.69	0.99	2.45	0.55
Direct Red 7	4.21	1.89	4.21	3.50	3.21	3.23	2.12
Direct Black 22	4.50	0.98	0.98	1.70	3.89	1.92	1.25
Direct Blue 1	2.55	1.96	6.09	3.68	4.78	3.59	3.09
Vat Yellow 2	0.56	0.67	0.65	0.75	0.87	1.22	0.95
Vat Red 10	0.55	0.79	0.79	0.83	0.76	1.62	0.88
Vat Brown 1	0.53	0.72	0.42	0.90	0.68	1.32	0.87
Acid Yellow 17	8.55	8.55	7.31	6.86	4.43	8.22	9.62
Acid Orange 7	7.24	10.71	10.71	7.67	10.87	13.74	8.99
Acid Red 73	7.88	9.45	7.45	10.99	4.56	8.34	8.30
Disperse Orange 3	1.51	1.59	1.39	1.54	0.77	2.86	1.80
Disperse Orange 13	1.65	1.86	1.76	1.35	0.87	0.82	1.79
Disperse Red 11	1.53	1.25	1.25	1.70	0.96	1.69	2.58

Effect of SDS surfactant: Fabrics subjected to electrochemical method of polymerization followed by immersion in the dye solution have produced improved dyeability in majority of the systems studied than the unpolymerized fabrics dyed in the conventional dyeing method. In the electrochemical polymerization, the improved K/S values were noted when Py/SDS/TBATFB/dye system was used with and direct, disperse and vat dyes. In the case of reactive and acid dyes, Py/SDS/dye system (showed increased K/S values. Among the systems studied, dyeing in Py/SDS/dye system was more effective in the dyeability than other systems. Considering the dye uptake and washing fastness, acid dyes produced better dyeability when compared to other dyes studied. Among the acid dyes, AO 7 has resulted better dyeability than other two.

Effect of CTAB surfactant: In the electrochemical polymerization followed by immersion in the dye solution, improved K/S values were noted when Py/CTAB/TBATFB/dye system was used for all classes of dyes. Considering the dye uptake and washing fastness, acid dyes produced better dyeability when compared to other dyes studied.

Among the acid dyes, AO 7 has resulted improved dyeability. Polymerization of pyrrole in the presence of surfactants such as dodecylbenzyl sulphonic acid or sodium dodecyl sulphate led to an increase in mass yield due to the incorporation of surfactant into the polymer. This is in agreement with the literature report (Stejskal *et al.*, 2003). Among the surfactants studied, CTAB has resulted better dyeability than SDS. Among the dopants studied, TBATFB has shown better dyeability than BMimTFB, which is supported by the increase in crease recovery angle values. But the use of ionic liquid (BMimTFB) resulted in the alteration of morphology in accordance with previous report (Pringle *et al.*, 2004). In electrochemical polymerization method, the monomer molecules are oxidized at the anode and these in turn attract the other monomer molecules leading to polymer formation on the electrode. The coloration of cotton and silk fabrics was higher when dopant was used. This could be due to the improved electrostatic attraction between dopant and free hydroxyl groups in cotton and -NH₂ group in silk, thus fixing the dyes in the fabrics accordance with previous report (Nouri *et al.*, 2000). Silk and polyester fabrics have shown poor electrical conductivities, which could be due to poor doping during polymerization. The conductivity results were obtained for cotton, silk and polyester fabrics. Significant conductivity was observed for cotton only; better conductivity

Table 3 Comparison of chroma values in different coloration systems

Dye	Chroma values						
	Conventional	Py / SDS / dye	Py / SDS / TBATFB / dye	Py / SDS / BMimTfB / dye	Py / CTAB / dye	Py / CTAB / TBATFB / dye	Py / CTAB / BMimTfB / dye
Reactive Blue 4	6.03	1.66	4.85	4.52	4.33	11.01	5.44
Reactive Orange 4	22.49	10.90	11.49	19.21	21.30	37.11	19.59
Reactive Violet 5	6.12	6.39	7.26	5.20	10.55	24.11	6.29
Direct Red 7	39.55	15.76	33.03	22.96	17.78	32.44	26.72
Direct Black 22	5.48	0.93	2.97	3.15	3.54	4.44	2.52
Direct Blue 1	25.07	16.38	35.22	24.68	22.61	33.33	29.92
Vat Yellow 2	14.87	11.11	5.46	14.00	15.65	22.51	18.77
Vat Red 10	15.79	11.32	13.10	12.24	12.65	15.03	15.11
Vat Brown 1	9.23	7.34	6.19	7.07	6.45	9.47	7.91
Acid Yellow 17	35.63	2.38	6.03	53.19	4.78	26.18	7.26
Acid Orange 7	46.02	17.45	58.29	50.32	35.65	64.29	46.59
Acid Red 73	44.97	14.01	43.05	54.87	33.89	45.88	43.14
Disperse Orange 3	23.33	15.77	17.55	12.73	15.21	14.39	11.93
Disperse Orange 13	15.83	25.32	20.08	9.30	3.55	7.74	9.99
Disperse Red 11	22.52	22.20	15.81	20.90	10.89	12.95	12.31

Table 4 Dyeing of silk with Py/CTAB/TBATFB/dye system

Dye	CCM data for dyed fabric						CCM data for dyed fabric after washing					
	L*	a*	b*	C*	H	K/S	L*	a*	b*	C*	H	K/S
AY 17	69.19	-4.81	25.73	26.18	100.60	9.35	71.15	1.63	5.58	5.81	73.70	8.22
AO 7	52.51	35.30	53.74	64.29	56.70	15.96	66.05	16.44	19.66	25.62	50.10	13.74
AR 73	61.54	43.62	14.23	45.88	18.06	10.36	61.80	27.10	2.49	27.21	5.25	8.34

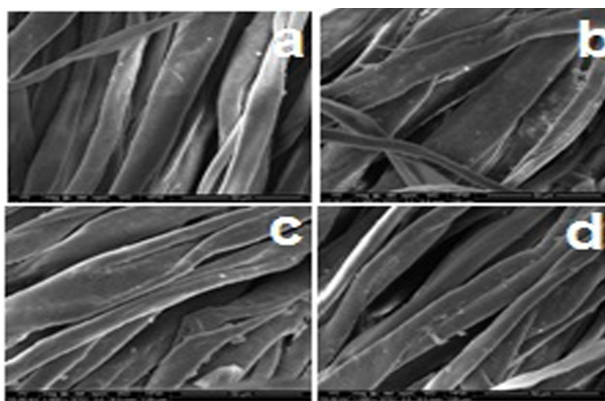


Figure 1 SEM images of colored fabrics, (a) Cotton with Py/SDS/TBATFB/DB1 system, (b) Cotton with Py/CTAB/TBATFB/RV5 system, (c) Cotton with Py/CTAB/BMimTfB/VR10 system and (d) Polyester with Py/SDS/DIO13 system

of 1.24×10^{-6} S/cm was obtained with DB 1 dye in Py/SDS/TBATFB /dye system. Kaynak *et al.*, 2009 studied the effect on the incorporation of fluorescent dopants into the polymerization solution as well as in the encapsulation of fluorescent dyes in a polypyrrole (PPy) micelle. It was determined on the basis of conductivity and fluorescent measurements that the encapsulation of dyes in PPy onto the surface of textiles gave the best results. In this study also, better conductivity was observed.

SEM images showed the nature of polymer coating on the fabric, which reveal the deposition of polypyrrole on to the fibres. The surface morphology of the fabrics obtained by SEM analysis was found to be better with (i) Py/SDS/TBATFB/DB1 system

- (ii) Py/CTAB/TBATFB/RV5 system
- (iii) Py/CTAB/BMimTfB/VR10 system
- (iv) Py/SDS/DIO13 system (Figure 1).

CONCLUSION

The polymerized fabrics showed improved dye attachment than conventional dyeing. The advantage in this proposed method is that conductive fabrics could be colored effectively by electrochemical polymerization, but without the use of dye auxiliaries and heating. This approach is a novel method of coloration on conductive textiles.

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