



## Hair Coloration Using Functionalised Polyethyleneimine/Dye Complex

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**A** new concept of colouring human hair fibres by coating them with a dyestuff coupled to a carrier (a positively charged polymer) is introduced and evaluated. The complex dyestuff-charged polymer is synthesised and applied on hair under normal conditions. The polymer-carrier, used in this work, is the branched poly(ethylene imine) (PEI), whose primary amine groups were modified at different ratio with quaternary ammonium groups and synthesised reactive dyestuff to give polymer-dye conjugates. The adsorption of charged substances from external aqueous media to hair has been investigated under neutral pH. The colour covers evenly the hair fibre and proves to be fast to shampooing, although the polymer-dyestuff complex is confined to cuticle region only. Compared to reference, the coating of hair yields higher colour intensity than the infiltration of the fibre.

**Keywords:** Polyethyleneimine, Hair Coloration, Dye complex.

### Introduction

Since the antiquity, hair has been dyed with natural dyes and then with synthetic ones as chemistry was rising [1]. Hair dyeing is nowadays a main concern for cosmetology because of growing care taken in self-appearance by both woman and men [2-5]. There are three ways to modify the colour of hair: it may be made lighter by bleaching; artificial colours may be added to the hair, or a combination of both of these methods may be employed.

Taking into account the durability at washing hair coloration is classified into three major categories: temporary, semi-permanent and permanent. Temporary dyes or colour rinses, do not bind firmly, and may be removed by single shampooing [6-8]. Semi-permanent products

consist of dyes that diffuse into and bind temporary to the hair sites. Because these dyes are not firmly bound, they diffuse out of the hair after a few shampoos [9-12]. As hair coloration is nowadays mainly used to cover grey hair the permanent coloration effect is required.

Permanent hair dying is different from the first two mentioned methods. Small colourless or slightly coloured dye precursors are deposited on hair surface, diffuse and migrate into hair and are oxidized by hydrogen peroxide to active intermediates [12-14]. The active intermediates react further inside hair with colour couplers to provide shampoo-resistant hair dyes. The dye is therefore confined in the fibre for a long time. However, although the customer likes long lasting effect of permanent colour, the damage that it causes to hair is a major drawback.

This study examines an alternative new idea of permanent hair coloration with less damage of the hair structure. The colour is achieved by coating the hair surface with the dyestuff coupled to a positively charged polymer-carrier [15]. The general principle is presented in Figure 1. [16]

Cationic polymers are largely used for hair care products as conditioning agents. It is known that these polymers are highly substantive to hair under mild conditions, because the hair surface bears a negative charge at pH above its isoelectric point of 3.61 [17, 18]. As an example, the adsorption of cationic hydroxyl cellulose onto hair occurs about 50 times faster than of the uncharged polymer [19].

The electrostatic interaction between cationic groups of polymer and anionic charge existing on hair surface is responsible for the polymer coating and for the durability at shampooing. As colouring product is linked to the polymer adsorbed on the hair surface, it does not diffuse into hair cortex and the results are less damaging hair structure.

## Experimental

### Materials

Glycerol (Acros), diethyl carbonate (Acros), 1,4-diazobicyclo[2.2.2]octane (DABCO, Aldrich), pyridine (Aldrich) phenyl chloroformate (Acros), 3-dimethylamino-1-propylamine (Aldrich), methyl iodide (Acros), Na<sub>2</sub>SO<sub>4</sub> (Acros),

poly(ethylene imine) (PEI, water free  $M_w=25000$  g/mol, Aldrich), dichloromethane (Acros), toluene (Acros) and *N,N*-dimethylformamid (Acros) were used as received. Tetrahydrofuran (Fluka) – THF – was dried over sodium and distilled before use.

Tresses of Caucasian blond and brown hair have been acquired from Kerling and used for colouring after a light shampooing for cleaning.

### Synthesis

#### *2-oxo-1,3-dioxolan-4-yl)methyl phenyl carbonate (1).*

Glycerol (10 g, 109 mmol), dimethyl carbonate (29.2 g, 326 mmol) and DABCO (121 mg, 1.08 mmol) were mixed and heated at 75°C for 10 h. After distillation of formed methanol and excess of dimethyl carbonate obtained glyceryl carbonate was dissolved in dichloromethane and pyridine. The solution was cooled to 0°C, and phenyl chloroformate was added drop-wise to keep the temperature below 5°C. Reaction was stirred overnight at room temperature. Formed pyridine hydrochloride was removed by filtration and the solution was washed three times with water and brine. Organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and dichloromethane was evaporated to give white powder of (2-oxo-1,3-dioxolan-4-yl)methyl phenyl carbonate. The product was further purified by recrystallization from toluene. <sup>1</sup>H NMR and <sup>13</sup>C NMR corresponded to the data reported in the literature [20].

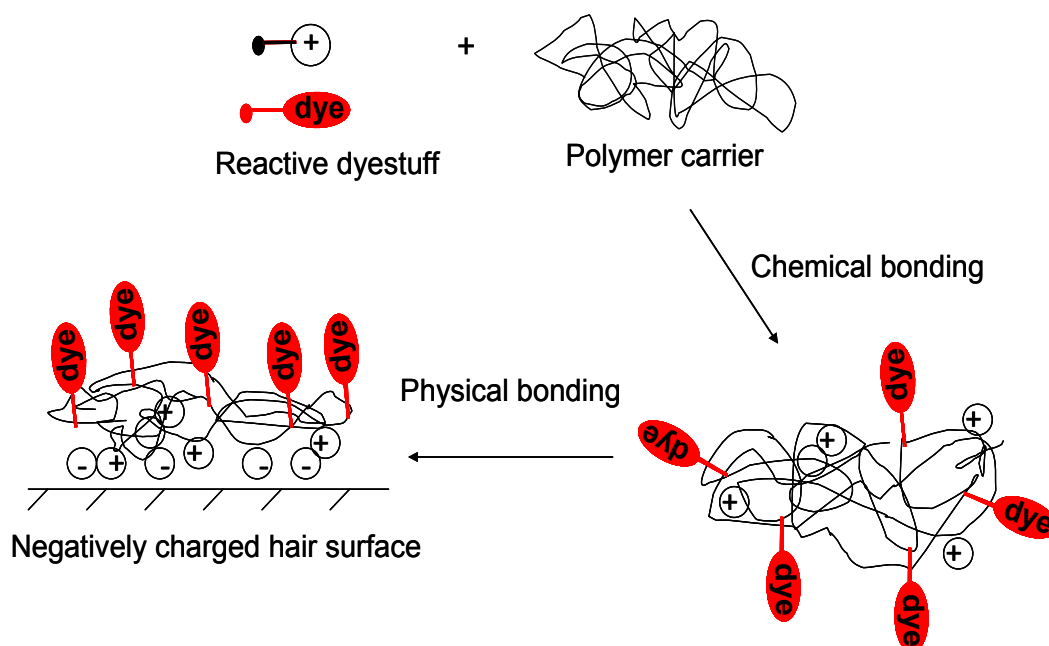


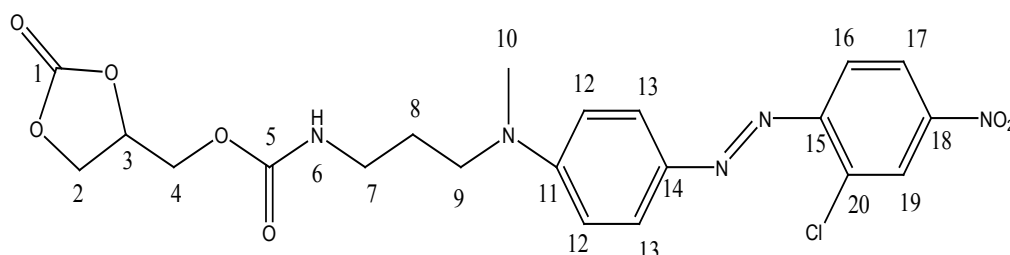
Fig. 1. Coloration of hair using polymer-dye conjugates.

3-*N,N,N*-Trimethyl-(((2-oxo-1,3-dioxolan-4-yl)methoxy)carbonylamino)propan-1-aminium iodide (QI).

Dicarbonate 1 was dissolved in dry THF and cooled to 0°C. The solution of 3-dimethylamino-1-propylamine in dry THF was added drop-wise and temperature was kept below 5°C. The reaction mixture was stirred for 20 h and next heated to 60°C. A solution of methyl iodide in dry THF was added drop-wise under intensive stirring within 3 h and the solution was left for additional hour at 60°C. Formed white power was filtered and dried under vacuum. <sup>1</sup>H NMR and <sup>13</sup>C NMR corresponded to the data reported in the literature [20].

2-oxo-1,3-dioxolan-4-yl)methyl 3-((4-((2-chloro-4-nitrophenyl) diazenyl) phenyl) (methyl) amino) propylcarbamate - reactive dyestuff (RD).

2-oxo-1,3-dioxolan-4-yl) methyl phenyl carbonate was dissolved in dry THF and the solution was cooled to 0°C. Next the solution of amine in dry THF was added drop-wise so that the temperature does not overcome 5°C. The reaction mixture was stirred at RT for additional 20 h. The solvent was removed by distillation (40°C, 30 mbar). The raw product dissolved in CH<sub>2</sub>Cl<sub>2</sub> was extracted with water, NaOH (5%), (HCl 5%) and brine to give (after evaporation of solvent) yellow oil.



Scheme 1. Reactive dyestuff (RD)

<sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>): δ= 1.68-1.82 (m, 2H, H<sup>8</sup>); 2.95-3.20 (m, 5H, H<sup>10</sup>, H<sup>7</sup>); 3.43-3.60 (t, 2H, H<sup>9</sup>); 4.15-4.40 (m, 3H, H<sup>4a</sup>, H<sup>2</sup>); 4.50-4.65 (t, 1H, H<sup>4b</sup>); 4.95-5.10 (m, 1H, H<sup>3</sup>); 6.80-6.95 (d, 2H, H<sup>12</sup>); 7.45-7.55 (m, 1H, H<sup>6</sup>); 7.70-7.80 (d, 1H, H<sup>16</sup>); 7.80-7.90 (d, 2H, H<sup>13</sup>); 8.15-8.25 (d, 1H, H<sup>17</sup>); 8.35-8.45 (d, 1H, H<sup>19</sup>).

<sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>): δ= 26.7 (C<sup>8</sup>); 38.08(C<sup>10</sup>); 38.36(C<sup>7</sup>); 49.37(C<sup>9</sup>); 63.15(C<sup>2</sup>); 65.88 (C<sup>4</sup>); 74.84 (C<sup>3</sup>); 111.70 (C<sup>12</sup>); 117.92 (C<sup>16</sup>); 123.30 (C<sup>17</sup>); 125.68 (C<sup>19</sup>); 126.61 (C<sup>13</sup>); 132.28 (C<sup>20</sup>); 143.29 (C<sup>14</sup>); 146.65 (C<sup>15</sup>); 152.30 (C<sup>18</sup>); 152.98 (C<sup>11</sup>); 154.69 (C<sup>1</sup>); 155.68 (C<sup>5</sup>).

#### Formation of polymer-dye conjugates

1 g of poly(ethylene imine) PEI was dissolved in 10 mL of DMF. The mixture of functionalized dyestuff and QI with desired ratio (see Table 1) was dissolved in 20 mL of DMF and added to the PEI solution. The reaction mixture was immersed in the oil bath at 60°C. After 48 h DMF was removed by evaporation under reduced pressure to give intensively red powder. The prepared modified PEIs were used without further purification to the coloration of hair. [21]

#### Applying procedures

##### Coloration of hair

The colouring was performed with 0.2 g/L water solutions of modified poly(ethylene imine)s

at 35°C and pH 6-7. The tresses of about 4 g of hair (liquor ratio of 1:50 - 1 g of hair for 50 mL of polymer solution) were immersed in polymer solution and left for 30 min. After that time hair were taken out of the solution, dried and used for further analysis.

##### Resistant of polymer layer for shampooing

In order to check the strength of the binding of modified poly(ethylene imine) to the hair surface the coloured hair were shampooed for 3 min at 40°C using 0.5ml/L shampoo (sodium lauryl ether sulfate 15%, pH 5.5), followed by rinsing with cold water for 90 sec. The procedure was repeated for three times.

**TABLE 1. Reagent ratio**

|        | $m_{PEI}$ [g] | $m_{OI}$ [g] | $m_{RD}$ [g] |
|--------|---------------|--------------|--------------|
| FPEI 1 | 1             | -            | 1.15         |
| FPEI 2 | 1             | 0.16         | 0.92         |
| FPEI 3 | 1             | 0.33         | 0.69         |
| FPEI 4 | 1             | 0.51         | 0.46         |
| FPEI 5 | 1             | 0.67         | 0.23         |

### Measurements

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on Bruker DPX-300 FT-NMR spectrometer at 300 MHz and 75 MHz, respectively. DMSO- $d_6$  was used as solvent. UV-VIS spectra were recorded on the UV-160A spectrophotometer (Shimadzu) in the scanning wavelength from 200 to 900 nm at concentration of modified poly(ethylene imine)s in water 0.02 g/L. UV-VIS measurements were also applied to quantitatively estimate the amount of adsorbed modified poly(ethylene imine)s onto hair. The change of solution extinction before and after coloration (diluted 10 times), at  $\lambda = 439$  nm was followed. Colour intensity achieved on hair was measured with Data Color 200 equipment. For following the penetration of dye into hair structure cross-sections of hair were examined with Aristoplan optic microscope.

## Results and Discussion

### Synthesis of reactive dyestuff

Reactive dyestuff was obtained in two-step reaction using (2-oxo-1,3-dioxolan-4-yl)methyl phenyl carbonate as starting compound. Pasquier *et al.* [20] shown that this type of bicarbonate reacts chemoselectively with primary amines. At low temperatures ( $0^\circ\text{-}25^\circ\text{C}$ ) phenyl ester carbonate reacts to give as side product phenol, while cyclic carbonate ring is opened only at higher temperatures ( $25\text{-}60^\circ\text{C}$ ).

Based on these results in the first step of dyestuff preparation the reactive cyclic carbonate ring was introduced to the structure of methylphenylamine propyl amine as presented in Scheme 2. After removal of side product (phenol), chromophore moiety was introduced to the benzene ring using azo-coupling reaction to give reactive dyestuff.

The resulting reactive azo-dye substance was a red powder soluble in organic solvents like DMF, *J. Text. Color. Polym. Sci.* **Vol. 19**, No. 1 (2022)

THF or chloroform but insoluble in water. UV-VIS measurements, plotted in Figure 2, shows the absorption band in the range 400 – 600 nm with a peak at 505 nm, typical for red dyeing dyestuffs [21].

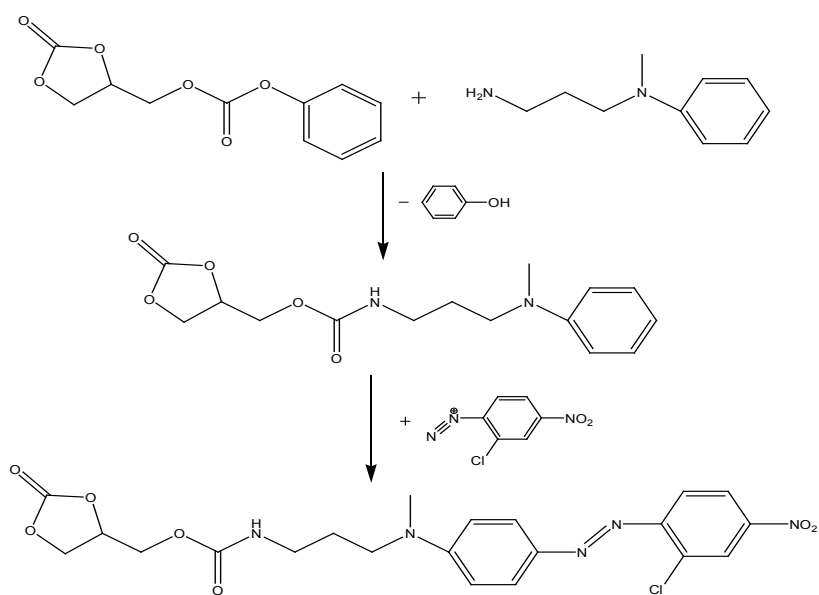
### Synthesis of modified poly(ethylene imine)s

Poly(ethylene imine) (PEI) of  $M_w = 25\ 000$  g/mol was chosen as polymer-carrier. It is a commercially available branched polymer bearing about 25 mol-% of primary amine groups, 50 mol-% of secondary amine groups and 25 mol-% of tertiary amine groups. The presence of high amount of amine groups enables its modification and introduction of high amount of different functionalities [16]. However, as cyclic carbonate ring introduced to the dyestuff structure reacts only with primary amines we were able to modify only 25 mol-% of repeating units of PEI.

The mixture of cationic coupler and non-ionic dyestuff in different ratio (see Table 2) was used to get the polymer-dye conjugates with different content of dyeing substance and cationic charge. [21]

The structure of the obtained polymer-dye conjugates was investigated by  $^1\text{H}$  NMR and UV-VIS measurements. The coupling of PEI with the functional carbonates is supported by the appearance of new signals, which can be assigned to formed urethane bonds. Additionally, the characteristic signals of the functional cyclic carbonates disappeared as shown in Figure 3.

The composition of resulting products could not be calculated from the spectra because of the overlapping of signals from dyestuff and cationic segment. However, as full conversion of reactive moieties was obtained the composition of the resulting modified poly(ethylene imine)s should equal the theoretical one. It is important for further experiments that the synthesised



Scheme 2. Synthesis of reactive dyestuff

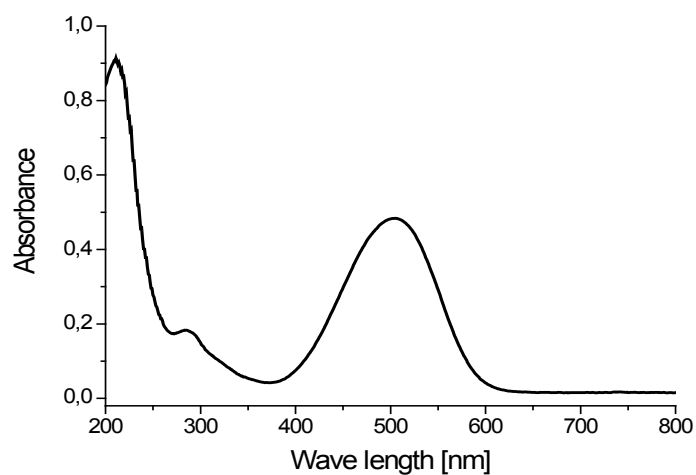
Fig. 2. UV-VIS absorption spectra of reactive dyestuff in THF ( $c = 0.02$  g/L)

TABLE 2. Composition of modified poly(ethylene imine)s

|        | mol-% of functionalized dyestuff * | mol-% of cationic functionalized coupler * |
|--------|------------------------------------|--|
| FPEI 1 | 100                                | -  |
| FPEI 2 | 80                                 | 20   |
| FPEI 3 | 60                                 | 40   |
| FPEI 4 | 40                                 | 60   |
| FPEI 5 | 20                                 | 80   |

\* refers to amount of primary amine in poly(ethylene imine) structure.

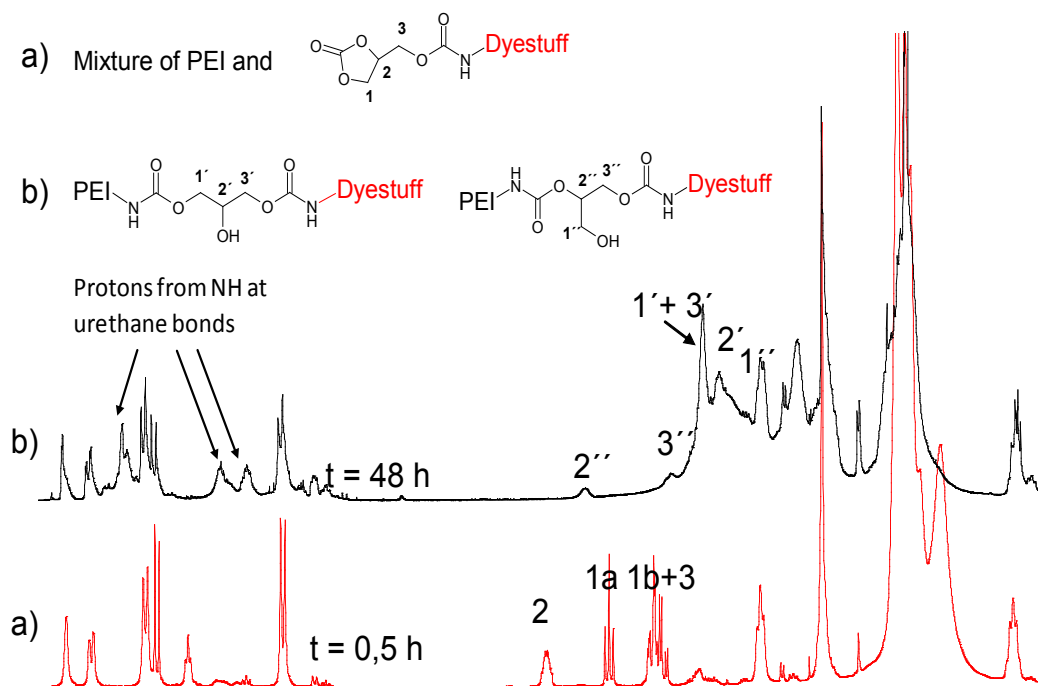


Fig. 3.  $^1\text{H}$  NMR spectrum of modified PEI in  $\text{DMSO-}d_6$  (FPEI 1)

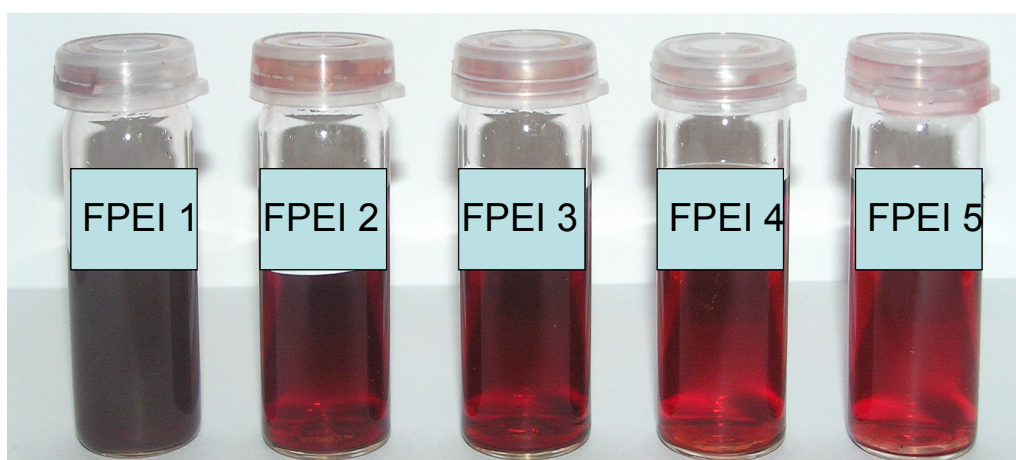
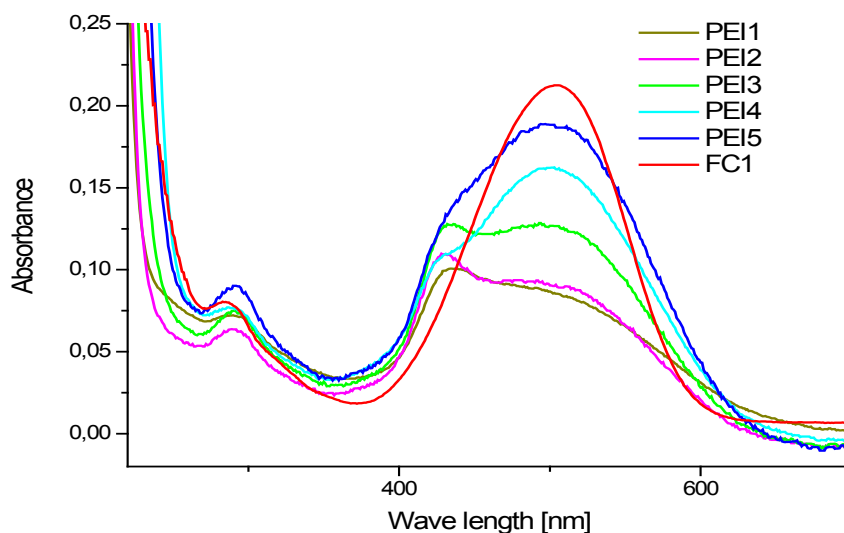


Fig. 4. Water solutions of polymer-dye conjugates at  $c = 0.2 \text{ g/L}$ .

polymer-dye conjugates are soluble in water. As it can be seen in Figure 4 the intensity of the red colour of water solutions of polymer-dye conjugates increases as expected with increasing amount of dyestuff in the polymer structure (at the same concentration of polymer-dye conjugate in solution). Additionally it confirms the

formation of covalent urethane bonds between amine groups of polymer and carbonate ring of dyestuff. The solubility of formed polymer-dye conjugates derives from highly hydrophilic PEI structure as dyestuff is insoluble in water. [16, 21]

The UV-VIS spectra of investigated polymer-



**Fig. 5. UV-VIS spectra of modified poly(ethylene imine)s in water at concentration of dyestuff 0.02 g/L; Functionalized dyestuff was measured in THF at  $c = 0.02$  g/L.**

dye indicates the hypsochromic shift of the band characteristic for dyestuff from 505 nm to 435 nm. This phenomenon was more pronounced for samples with higher amount of introduced dyestuff. As the result colour of sample solutions with high content of dyestuff is purple-red, while it is red for the samples with lower content of dye (40 mol% and less). It should be noted that samples for UV-VIS measurements were prepared in that manner that the amount of dyestuff in the solution was constant (concentration of polymer-dye conjugate was varied). Nevertheless, the intensity of the peak deriving from dyestuff is much lower for samples with high amount of dyestuff in its structure (FPEI 1 and FPEI 2).

#### *Coloration of hair*

The results of coloration were examined both for exhaustion with spectrophotometer (on solution used for coloration), and for colour intensity (spectrophotometer with reflection and visual). Three samples of polymer-dye complex were used for dyeing hair at neutral pH. Two of the samples (FPEI 3 and FPEI 5) possess cationic charge introduced by reaction with functional coupler QI, while FPEI 1 has only the dyestuff incorporated into PEI structure. Although PEI was not quaternized by introduction of QI segments, its water solution is charged because a large number of its amine groups is protonated even at neutral pH [22]. The tresses of hair before and after coloration are shown in Figure. 6.

As it can be seen the strong change of the colour between blond reference and dyed hair appeared for all applied polymer-dye conjugate samples used for coloration. The colour intensity measured with Data Colour, gave values of  $\Delta E$  in the range from 14 to 16 (Table 3). Also in case of brown hair the effect of coloration could be good visible, but as the change of the colour was not as drastic as for blond hair, the obtained  $\Delta E$  values varied at around 3.

The absorption observed for the polymer-dye conjugate solution after coloration was about 50 % lower than the absorption of initial sample. It means that about 50 % of polymer-dye conjugate was adsorbed on hair surface. In all cases the colour was distributed uniform on the hair surface without spots. The highest colour intensity was observed for the hair coloured with the solution of the polymer-dye conjugate sample without cationic groups, but containing the highest amount of dye in its structure (FPEI1).

In order to investigate the strength of polymer bind to hair surface, the tresses were shampooed and examined for the amount of colour removed by the help of extinction and Data colour measurements. The results listed in Table 3 show that the extinction of the solution from shampooing was negligible and only small decrease of the colour intensity measured with



Fig. 6. Hair tresses before (reference) and after dyeing with the polymer-dye conjugates.

TABLE 3. Colour intensity data from Data Colour measurements.

| Hair Type | FPEI | $\Delta E^*$     |                  |                   | Extinction       |                  |
|-----------|------|------------------|------------------|-------------------|------------------|------------------|
|           |      | After coloration | After shampooing | Before coloration | After coloration | After shampooing |
| Blond     | 1    | 16.1             | 15.9             | 0.874             | 0.440            | 0.011            |
|           | 3    | 14.1             | 13.0             | 0.555             | 0.294            | 0.008            |
|           | 5    | 13.9             | 12.8             | 0.244             | 0.136            | 0.007            |
| Brown     | 1    | 3.5              | 2.4              | 0.874             | 0.575            | 0.011            |
|           | 3    | 3.1              | 2.5              | 0.555             | 0.352            | 0.008            |
|           | 5    | 2.6              | 1.3              | 0.244             | 0.13             | 0.007            |

\*  $\Delta E = E_{\text{sample}} - E_{\text{reference}}$

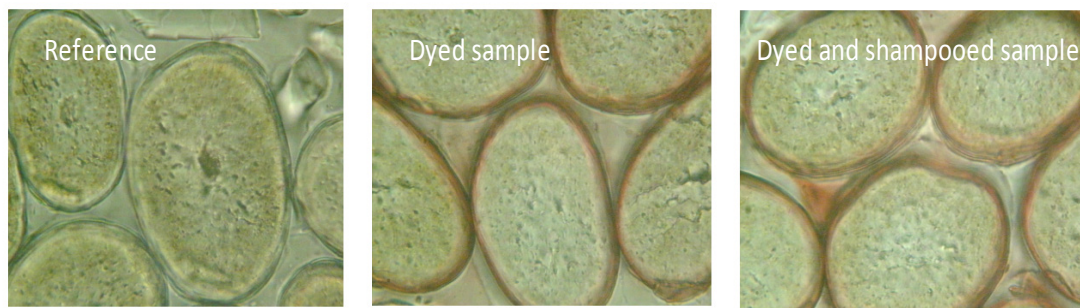


Fig. 7. Cross-sections of hair fibres before and after dyeing with polymer-dye conjugate (FPEI 1).



Data Colour was noticed. It means that only small amount of polymer-dye conjugate was washed out from the hair surface and confirms the efficiency of interaction between cationic polymer-peptide conjugates and hair. It can be also noticed, by comparing the results for the three FPEI polymer-dye complexes that the introduction of cationic groups to PEI structure is unnecessary as it seems that the polymer itself is charged enough to adsorb strongly on the hair structure.

Figure 7 shows the optical microscopy results of examining the cross-sections of hair fibres before and after coloration as well as after shampooing. It can be seen that the colour is confined to the cuticle and no penetration into deeper layers can be observed. The examination of the cross-section of the shampooed hair supports the results from Table 3, not showing any significant change of cuticle colouration (see Figure 7). This indicates on one side that all the dyestuff is bonded to the polymer, and on the other that the carrier adheres strongly to cuticle.

### Conclusions

We developed a new concept of permanently attaching dyestuff to hair by using a polymer-carrier. The route for the synthesis of the new reactive dyestuff, bearing cyclic carbonate ring, is detailed. It is coupled to a cationic coupler and is successfully used for preparation of charged PEIs (polymer-dye conjugates) applied further for hair coloration. The polymer-dye conjugate adsorbs on the anionic hair surface by the help of the cationic groups present in the polymer structure. Compared to reference significant change of hair colour was observed in case of all applied samples at low amount of dye used. Additionally, the colour was uniformly spread on the whole surface of hair. The confinement of polymer-dye conjugate to cuticle region was proved by cross-section images.

The adsorbed polymer-dye conjugate withstands shampooing, supporting the assumption of bonds created between amino groups of the polymer and carboxyl groups of the hair surface. Summing up, it appears possible to achieve a fast and good colouring yield at hair coloration by using a cationic polymer-carrier to bind reactive dyestuff molecules to fibre surface.

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<sup>٢</sup> المركز القومي للبحوث (رقم إنتساب Scopus ٦٠٠١٤٦١٨)، قسم بحوث الصناعات النسيجية، التحضيرات والتجهيزات للاليف السليلوزية، ٣٣ شارع البحوث (شارع التحرير سابقاً)، الدقي، ص. ١٢٦٢٢، الجيزة، مصر.

<sup>٣</sup> شركة هنكل ألمانيا.

تم تقديم مفهوم جديد لتلوين ألياف الشعر البشري عن طريق تغليفه بصبغة مرتبطة بحامل (بوليمر موجب الشحنة) وتقييمها. يتم تصنيع بوليمر الصبغة المعقد ذو الشحنة المحمل على البوليمر وتطبيقه على الشعر في الظروف العادية. مادة البوليمر الحاملة، المستخدمة في هذا العمل، هي البوليمر المتفرع (إيثيلين إيمين) (PEI)، والتي تم تعديل مجموعات الأمين الأولية الخاصة بها بنسب مختلفة مع مجموعات الأمونيوم الرباعية والصبغة النشطة المركبة لإعطاء اتحادات صبغة البوليمر. تم فحص ادمصاص المواد المشحونة من الوسائط المائية الخارجية إلى الشعر تحت درجة الحموضة المحايدة. يغطي اللون ألياف الشعر بالتساوي ويثبت أنه ثابت عند غسل الشعر بالشامبو، على الرغم من أن مركب صبغة البوليمر المعقد يقتصر على منطقة البشرة فقط. مقارنة بالمرجع، ينتج عن تلوين طبقة الشعر السطحية كثافة لونية أعلى من تلك المتغلطة داخل الألياف.