



Synthesis, identification and application of aldehyde reactive dyes



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ABSTRACT

Nine aldehyde reactive dyes, designated **D**₁–**D**₉, were synthesized. Among them, **D**₁ and **D**₂ were synthesized by “grafting” alizarin red and alizarin yellow GG, respectively, to the α -C of n-butyl-aldehyde; **D**₃ and **D**₄ were synthesized by “grafting” alizarin red and alizarin yellow GG, respectively, to the methyl-C of 4-methyl-cinnamaldehyde; **D**₅–**D**₉ were synthesized by “grafting” alizarin red, alizarin yellow GG or self-prepared colorants to the α -C of glutaraldehyde. UV–Vis, FTIR, ESI-MS, elemental analysis and ¹H NMR were applied to identify the target products. Aluminum-tanned wet-white sheep leather was chosen as substrate for the application experiments, and their four dyeing property indexes, i.e., substantivity (*S*), exhaustion (*E*), fixation (*F*) and reactivity (*R*) were measured. The results indicated that, without adding of neutral salts and under mild conditions (*T* = 35–45 °C, pH = 4.0–6.5), all these dyes could dye wet-white leather within 1.5 h, and the dyed leather possessed a satisfactory washing-fastness.

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1. Introduction

Reactive dyes are widely used to dye protein fibers (such as wool, silk, fur and leather) and cotton fiber because of their good rub fastness and washing fastness. However, research results [1–3] showed that the problem of the currently available reactive dyes are: (1) the hydrolysis of their reactive groups during the process of storage and application results in the lost as high as 20–50% of reactive dyes; and (2) a large quantity of neutral salts (20–40 g/L) required in the dyeing process may cause environmental pollution.

The idea of reactive aldehyde dyes derived from the research of our laboratory on the color-forming mechanism of natural iridoids (such as genipin, loganin aglycone, oleuropein aglycone and E-6-O-methoxycinnamoyl scandoside methyl ester aglycone) with primary amino (–NH₂) containing materials (such as silk, leather and wool) [4,5]. This mechanism indicated that there was an inter-conversion isomer with a glutaraldehyde backbone present in the reaction system, which caused genipin, for example, to crosslink with collagen fiber (Fig. 1). Two effects would produce during this process: collagen fiber was dyed to dark-blue and its hydrothermal stability was increased [4,5].

Glutaraldehyde is a commonly used tanning agent for leather manufacture. During the tanning process a crosslinking reaction could occur between glutaraldehyde and collagen molecules through a Schiff base reaction. This mechanism suggested that a new kind of reactive dye could be made by “grafting” bio-friendly colorants to the backbone of glutaraldehyde. Unlike the normally used reactive dyes, this alternative reactive aldehyde dyes do not hydrolysis and neutral salts do not need during their application. Theoretically, a simultaneous tanning – dyeing process could be achieved when this novel kind of reactive dye is applied to leather.

Generally, glutaraldehyde is preserved as a 25%–50% (w/w) aqueous solution and it is almost impossible to “graft” colorant compounds to its backbone directly. So an “easy to difficult” strategy was adopted, during which colorant compounds were grafted to the α -C of n-butyl-aldehyde and the methyl-C of 3-(4-methyl) phenyl -acrylaldehyde, so that a series of reactive dyes with mono-aldehyde as reactive group was obtained. Their capacity to dye aluminum pre-tanned wet-white leather were investigated; after that, colorant compounds were grafted to the precursor of glutaraldehyde, and target products were obtained by an oxidation reaction or hydrolysis reaction. In this way, nine reactive dyes with aldehyde as the reactive group were synthesized. Their structures were identified and their application properties were investigated.

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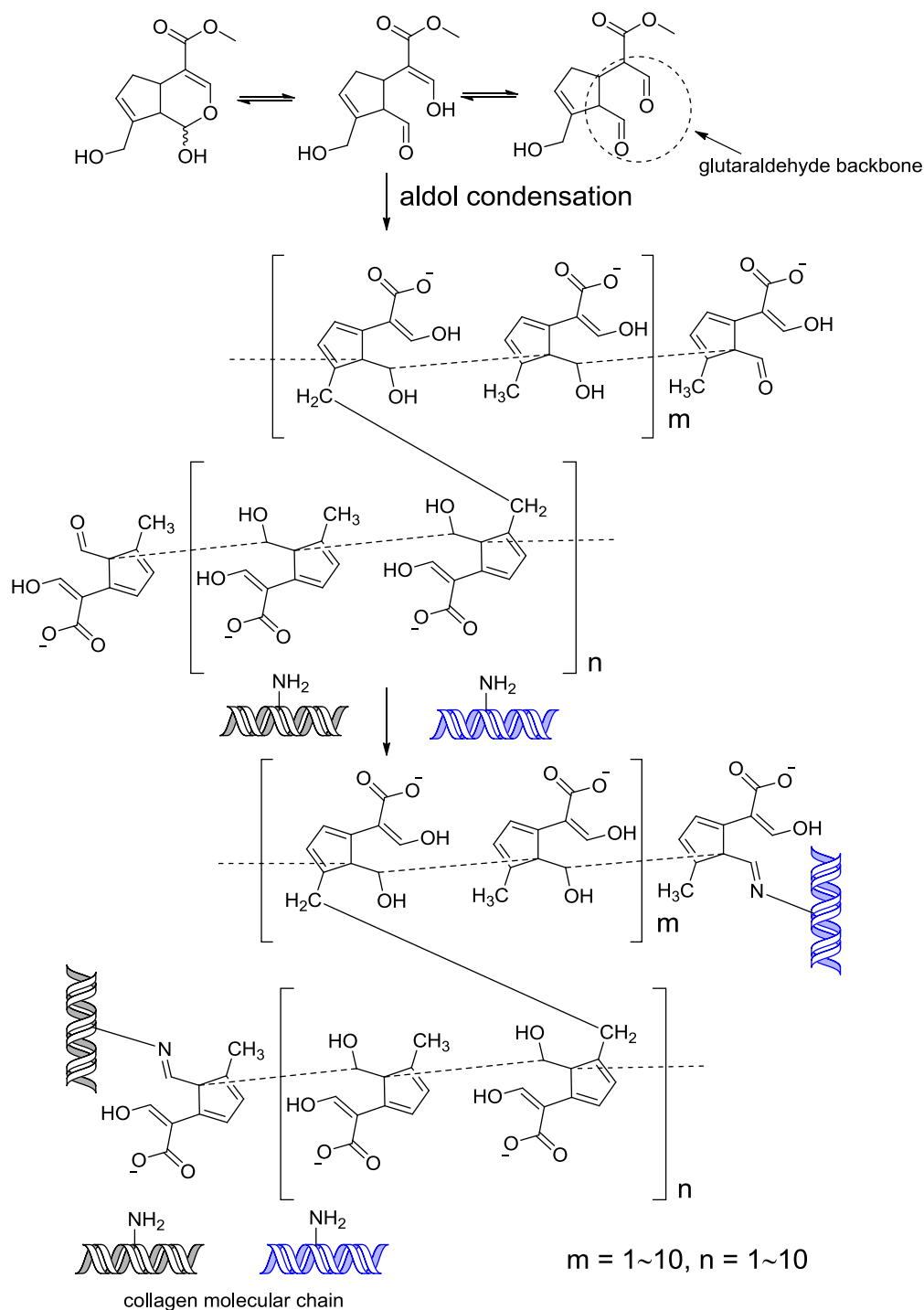


Fig. 1. The crosslinking and color forming mechanism of genipin with collagen fiber.

2. Experimental

2.1. Materials

3,4-Dihydro-2H-pyran-2-methanol ($\approx 97\%$) was supplied by Hua-chem Industry Ltd., Japan; sodium 1-amino-4-bromoanthraquinone-2-sulfonate ($\approx 89\%$) was supplied by Longsheng Dye Chemical Corp., Zhejiang, China; Silica GF254 was supplied by Qingdao Haiyang Chemical Corp., China; n-butyl-aldehyde, N,N-dimethylformamide

(DMF), N-chlorosuccinimide (NCS), N-bromosuccinimide (NBS), L-Proline, Alizarin red, Alizarin yellow GG, 4-methylcinnamaldehyde, Benzoyl peroxide (BPO), cyclopentene, dimethyl sulfoxide (DMSO), 1,4-dioxane, sodium 2,4-diamino-benzenesulfonate, m-phenylenediamine, hydroquinone and other commonly used chemicals were supplied by Chengdu Kelong Chemical Corp., China (all analytical grade); aluminum pre-tanned sheep wet white leather was provided by the Leather Department of Sichuan University, China.

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