

# ‘Naturalization’ of textile disperse dyes through glycoconjugation: the case of a bis(2-hydroxyethyl) group containing azo dye

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**Abstract**—A family of five strictly related glycoconjugated azo dyes (GADs), characterized by the presence of the same chromophore and a variable number (1–4) of deprotected hexose units, has been prepared by employing succinate bridges for connecting the azo dye and the sugar portions. The modulation of the hydrophilic portion determines the appreciable changes in the water solubility of GADs. In all the cases, however, hydrophobic fibres (polyester) were homogeneously dyed with GADs at temperatures lower than that used for original azo dyes, at atmospheric pressure, and avoiding the use of surfactants. Furthermore, GADs show an interesting multipurpose character leading to dyeing well also the natural fibres as, for instance, wool. The presence of a variable number of hexose units in the different GADs determines some changes in the colour intensity of dyed fabrics, but in all the cases an appreciable rubbing and water fastness were maintained.

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## 1. Introduction

Archaeological traces of dyed clothes have been found through the world, showing that ancient civilizations in America, in the Mediterranean area and in the Central Asia developed efficient dyeing techniques using natural dyestuffs prevalently obtained from plants, but also from insects and shellfish.<sup>1</sup> Until the second half of the 19th century, textile industry developed more and more sophisticated techniques obtaining an impressive level of quality in dyeing fabrics with natural dyestuff.<sup>1</sup> Many vegetable natural colours are constituted by different classes of chromophores O- or C-glycosylated with mono-, di- or oligosaccharide moieties. Exam-

ples are<sup>1</sup> the cartamine, a deep red glycoside from Safflower (*Cartamus tinctorius*), the blue indicotine from Woad (*Isatis tinctoria*), obtained by the hydrolysis of the colourless glycoside indacane, the ruberitric acid from Madder (*Rubia tinctorum*), a glycoside of shining red colour constituted from alizarine and the disaccharide primaverose. Starting from the second half of the 19th century, following the Industrial Revolution, natural dyes have been almost completely replaced by synthetic compounds, which are less expensive and much easily available. Nowadays, textile industries dispose off a huge number (about 10,000) of synthetic dyes,<sup>2</sup> produced by chemical industries throughout the world in an estimated overall production of 700,000 tons/year.<sup>2</sup>

Amongst the different classes of synthetic dyes currently used in the textile industry, the by far most common is constituted by the so-called ‘disperse dyes’, represented mainly by the azo and the anthraquinone

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families.<sup>2</sup> Industrial dyeing processes are generally carried out in aqueous solution and require several auxiliary chemicals, as for instance surfactants.<sup>3</sup> The decontamination of effluents is a difficult task because the type and the extent of contamination largely vary depending either on the fabric dyed or the class of dye used. Furthermore, an approach coming back to an extensive use of natural dyes extracted from plants or animals at present is not a realistic hypothesis, because of the high quantities of dyes required by the global market, the impact of the extraction processes and the general low affinity of natural dyes with fibres requiring the use of specific chemical auxiliaries.

A recently proposed<sup>4</sup> innovative type of textile dyes is based on the idea of mimicking the structure of natural dyes through the modification of commercial synthetic disperse dyes by their glycoconjugation. A first family of glycoconjugated azo dyes (GADs, Chart 1) has been obtained,<sup>5</sup> putting a succinyl bridge from the azo chromophore and a selected, nonanomeric position of easily available mono- or disaccharides (D-glucose, D-galactose and lactose). The GADs obtained by this ‘naturalization’ method showed interesting dyeing properties. More recently, a second series of GADs, having a diethereal linker, have also been proposed.<sup>6</sup> In all cases, the appreciable water solubility of the GADs permits the development of effective dyeing processes in water employing reduced amounts of dye, without the addition of surfactants or other additives, under mild conditions of temperature and pressure.<sup>4,5</sup> Furthermore, GADs acquire an unexpected multipurpose nature, allowing a large kind of different fabrics (wool, silk, nylon, polyester, acrylic, polyacetate and polyurethane) to be dyed,<sup>4,5</sup> opening thus the way to solve the challenging problems related to the efficient dyeing of fabric blends.

A remaining problem to be solved is related to the modulation of the hydrophilic pocket, that is, the sugar portion of GADs, in view of their application in industrial processes allowing the most favourable compromise between water solubility and colour fastness.

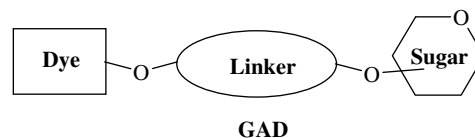


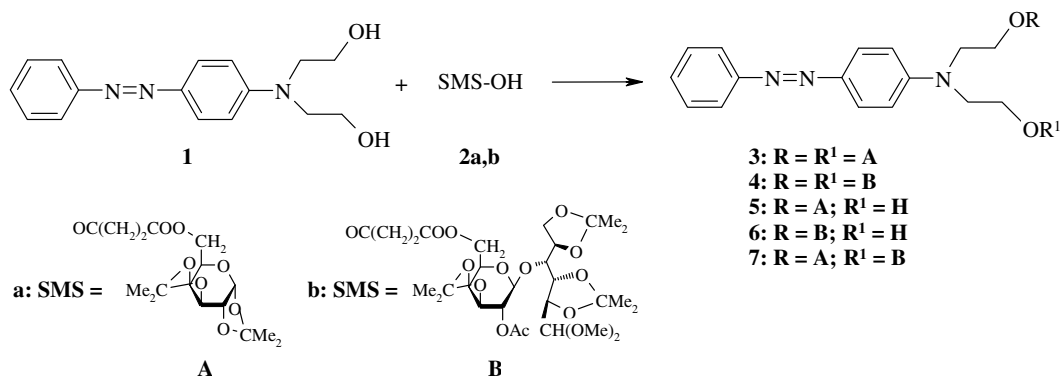
Chart 1. Schematic representation of a glycoazodye (GAD).

We are presenting now the synthesis of a family of five structurally related GADs obtained by the conjugation of one or two mono- or disaccharide units to the same synthetic yellow azo dye **1**,<sup>7</sup> characterized by the presence of a bis(2-hydroxyethyl) group exposing thus, two equivalent primary alcoholic functions (Scheme 1).

## 2. Results and discussion

### 2.1. Synthesis of glycoconjugated azo dyes (GADs)

The preparation of the protected bis-conjugated succinyl GAD **3** was easily achieved by the treatment of a THF solution of azo dye **1** and a slight excess (2.2 mol) of the known<sup>5</sup> protected sugar monosuccinate (SMS-OH) **2a** with *N*-(3-dimethylaminopropyl)-*N*-ethylcarbodiimide hydrochloride (EDC, 2.2 mol) in the presence of dimethylaminopyridine (DMAP). In accordance with our previous results on the conjugation of monohydroxy azodyes, the expected bis-succinate **3** was obtained in about 88% isolated yield. A very close result was obtained in the preparation of the analogous protected lactose GAD **4**. As expected, the preparation of mono-conjugated azo dyes **5** and **6** was much more complicated giving mixtures of mono- and bis-esterificated products and unreacted diol when a 1:1 molar ratio of dye and sugar monosuccinate was allowed to react. An acceptable yield of mono-conjugated GAD **5** was obtained (49% based on the sugar and 59% based on the reacted dye) by performing a slow addition at 0 °C of the sugar monosuccinate (**2a** and **2b**) to a solution containing an excess of dye **1** (1.5 mol **2a**) in the presence of EDC and DMAP. In an analogous manner,



Scheme 1. Synthesis of protected GADs.

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