

SAXS AND DSC STUDY OF MULTILAYER LAMELLAR VESICLES (MLV) IN HAIR DYE BASES

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INTRODUCTION

Multilayer Lamella Vesicles (MLV), as a very good delivery system for active ingredients, has been widely used in health care as well as in personal care products. MLV improves dispersion of difficult-to-solubilize compounds such as drugs and sunscreen oils; enhances adhesion on the skin surface and sustained release of active ingredients. Phosphate esters have long been used in hair and skin care formulations including hair relaxers, hair perms, sunscreens, and color bases as excellent emulsifiers and viscosity thickening agents. In previous studies (1), we reported that MLV structure formed in sunscreen formulas with a phosphate ester emulsifier played an important role enhancing the deposition of sunscreen oil on the skin surface, and therefore, improving the SPF water-wash resistance. It was also found that addition of phosphate esters into hair dye base formulas enhanced formation of multilayer lamellar vesicles (MLV) and improved their coloring performance. The molecular structure of added phosphate ester showed significant effects on the formation of MLV phases and rheological properties (2-3).

In this paper, we report our recent studies on phase structures of liquid crystals and phase transition temperatures of hair dye bases containing different phosphate esters by using Small Angle X-ray Scattering (SAXS) and Differential Scanning Calorimetry (DSC).

EXPERIMENTAL

- **Materials:** Three oxidative hair dye (auburn) bases (HCE, CS-20, and Ceteth-20) containing different phosphate ester/ non-ionic emulsifiers were prepared by Croda Japan. CS-20 base was made from Ceteth-20 Phosphate (and) Dicityl Phosphate; HCE base made from Oleth-5 Phosphate (and) Diolethyl phosphate; Ceteth-20 made from non-ionic emulsifier of Ceteth-20.
- **Instruments:** SAXS: NanoSTAR U from Bruker AXS Inc., Madison, WI
DSC: TA Q100 from TA Instruments, New Castle, DE
- **Experimental Set-up**
 1. SAXS: Sealed Tube X-ray source Cu-K α 40kV/35mA, focal spot = 0.4 mm X 8 mm; Bruker AXS HI-STAR position sensitive area detector; SAXS for Windows NT computer software. Color base samples were measured in a sample holder for gel like samples at ambient temperature (20°C). Kapton was used as window material.
 2. DSC: About 10 mg of color base sample was placed in a sealed pan and the heating rate was 2 degrees centigrade per minute

RESULTS AND DISCUSSION

1. Emulsion Structure of Color Bases

Figure 1 shows digital images of three color base samples under a microscope with crossed polarizers.

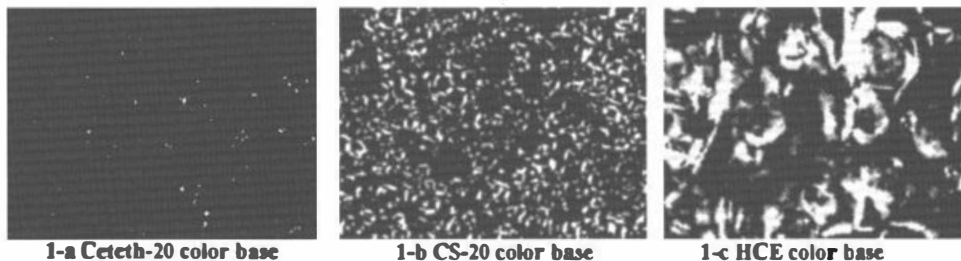


Figure 1 Micrograph of different color bases (X400)

It is seen that CS-20 and HCE color bases formed very clear MLV structure, and the determined average particle sizes are: Ceteth-20: 3.8 μm ; CS-20: 6.0 μm ; HCE: 12.5 μm . The average particle sizes are in the following decreasing order: HCE > CS-20 > Ceteth-20.

2. SAXS Patterns

Figure 2 explains SAXS 2D diffraction patterns of three tested color bases. Different SAXS patterns were observed on these bases. It was determined that the CS-20 color base exhibited hexagonal crystal phase with smaller average particle size, narrower size distribution, and larger space constant ($d \sim 124 \text{ \AA}$). The HCE dye base formed cubic crystal phase with larger average particle size, broader size distribution, and smaller space constant ($d \sim 95.7 \text{ \AA}$). It is interesting to note that Ceteth-20 color base formed a clear lamellar structure with the largest space constant of 146 \AA .

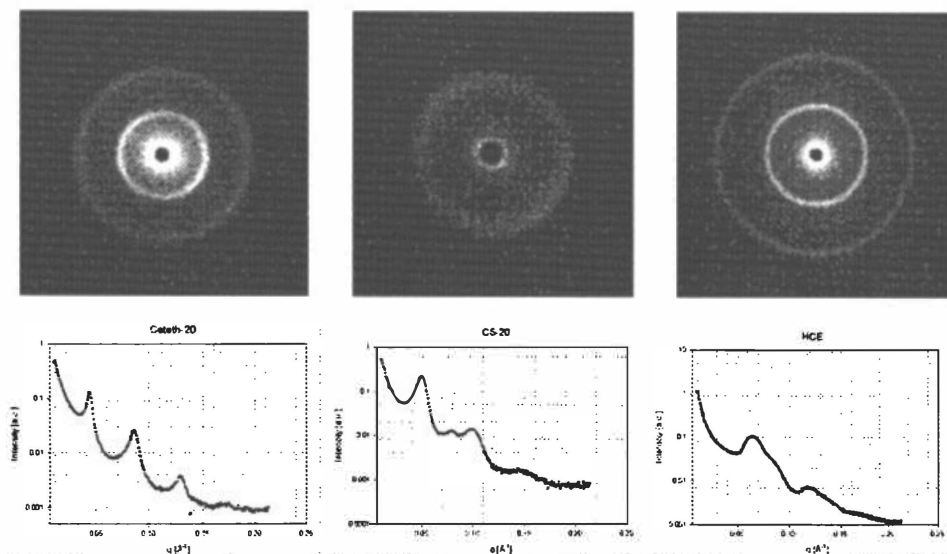


Figure 2 SAXS spectrum and diffraction patterns

3. DSC Study

DSC phase transition temperature and enthalpy are listed in Table 1.

Color Base Sample	Transition Temperature ($^{\circ}\text{C}$)	Transition Enthalpy (J/g)
Ceteth-20	62.2	6.24
CS-20	65.9	10.14
HCE	56.1	7.03

It can be seen that CS-20 dye base demonstrated higher phase transition temperature and larger transition enthalpy, which were corresponded to its well-organized liquid crystal structure, while HCE dye base showed lower phase transition temperature and smaller transition enthalpy.

References

1. T. Gao, J Tien and Y Choi, Sunscreen formulas with MLV structure, *C&T Magazine*, 118 (10), 41 – 48, 2003
2. T. Gao, PhD, A. Pereira, Y. Choi, J. Tien and R. Lanese. A Novel Phosphate Ester for Hair-coloring Enhancement, presented at the Annual Scientific Seminar of SCC, June, 2005, Las Vegas and published on *J. Cosmetic. Sci.*, 56, 374 -75, 2005.
3. T. Gao, PhD, R. Akatsuka, R. Bord, and A. Pereira, Multilayer Lamellar Vesicles in Oxidative Dye Formulations – Characterization and Performance”, *C&T Magazine*, 121(5). 75 – 88, 2006.

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