

Mechanisms of Spectral Sensitization of Silver Halides: Role of Sensitizing Dye Complexation*

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We have observed concurrent processes of photoexcited dye deactivation and silver(0) cluster formation in real time under conditions of laser flash photolysis of a merocyanine dye adsorbed to an AgBr nanosol. To our knowledge this is the first time such a comprehensive view of the process of spectral sensitization has been obtained experimentally. Spectral sensitization of AgBr is apparently biphotonic under our conditions; this result, along with observed kinetics of dye ground state repopulation and of silver(0) cluster growth, is consistent with Mitchell's mechanism of spectral sensitization, but not with single-electron transfer or radical pair mechanisms. Control experiments revealed a modicum of photolytic reactivity for undyed nanosol with 2.35 eV photons and suggested operation of an Auger mechanism of photoelectron generation. In this case amplified stimulated emission was observed from photogenerated silver clusters, Ag_n^0 (or a by-product) at photon energies comparable to those that produce the Hirschel effect in conventional photography. We speculatively estimate nuclearity of the silver clusters produced under conditions of our experiments as $n \approx \text{ca. } 12$.

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Introduction

The literature provides a number of mechanistic proposals with respect to the mechanism of spectral sensitization of photolysis of the silver halides for latent image formation in the photographic process. The most commonly cited mechanism¹ involves single-electron injection from a photoexcited, physisorbed dye molecule or aggregate to the conduction band of a silver halide microcrystal; this electron-transfer mechanism has its roots in early proposals of Gurney and Mott² on mechanism of latent image formation. It was later abandoned by one of its initial proponents,³ but then was revived and elaborated in the elegant work of Tani.⁴ Further definitive studies have related electrochemical potentials of dye molecules to sensitizing ability in the context of this model.⁵⁻⁸ In support of this mechanism, Chibisov and coworkers⁹ claim to have ob-

served radical dication products of imida- and oxocarbo-cyanine sensitizing dyes (as would result from single-electron transfer to the conduction band of AgBr from photoexcited dye) under conditions of laser flash photolysis of dyed AgBr photoemulsions. Note that the same radical dication products would have resulted if the adsorbed dye molecules had simply trapped photoholes from the AgBr valence band or surface states.

After an earlier suggestion by one of us,¹⁰ both the Muenter¹¹ and Tani¹² groups have shown that rate constants for electron transfer, k_{et} , presumably the key step in spectral sensitization of silver halide grains with a number of cyanine sensitizing dyes, can be correlated quantitatively with dye reduction potentials, using the Marcus model for single-electron transfer. These experiments, however, obtain k_{et} from fluorescence lifetime ($1/\lambda_p$) measurements, in which it is assumed that intersystem crossing, described by k_{isc} , is unimportant, i.e., $k_{isc} \ll (k_{et} + k_p)$. Recent picosecond- and nanosecond-resolved transient absorption spectroscopic studies on cyanine dyes in solution have shown that this may not necessarily be the case.¹³ It is furthermore equally appropriate to assign the process identified with k_{et} in this analysis to energy transfer.¹⁴

Fassler and colleagues¹⁵ have provided optical and electron spin resonance spectroscopic evidence that photoexcited dye adsorbed to a silver halide grain is reduced by an electron-donating surface state of AgBr (or a donor counterion), so that electron transfer to the silver halide conduction band occurs then from the resulting free radical species. These observations have led to the so-called radical pair mechanism of spectral sensitization.¹⁶ It can be contended, however, that this mechanism operates only in special cases, e.g., with a dye whose HOMO level is uniquely matched to that of the donor state¹¹ or under conditions of supersensitization.^{9,16}

After abandoning the electron-transfer model of spectral sensitization, Mott³ advocated an energy-transfer mechanism that presumed existence of otherwise undefined energy-accepting states located energetically within the forbidden gap of the AgBr host. This approach has been followed by Mitchell¹⁷ in his development of the "coupled electron transfer" model of spectral sensitization. Recent theoretical developments¹⁸ emphasize the continuity between energy transfer and coupled electron-transfer models. Because photon energy in spectrally sensitized photolysis is less than the dissociation energy for AgBr, it has been proposed in these models that spectral sensitization may be a biphotonic process.^{19,20} Mitchell has further proposed in this context that intrinsic, localized donor or acceptor centers within the AgBr crystals are crucial to

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Spectral Sensitization

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Table of Contents Spectral Sensitization

1. Understanding the eBook Spectral Sensitization
 - The Rise of Digital Reading Spectral Sensitization
 - Advantages of eBooks Over Traditional Books
2. Identifying Spectral Sensitization
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Spectral Sensitization
 - User-Friendly Interface
4. Exploring eBook Recommendations from Spectral Sensitization
 - Personalized Recommendations

- Spectral Sensitization User Reviews and Ratings
- Spectral Sensitization and Bestseller Lists
- 5. Accessing Spectral Sensitization Free and Paid eBooks
 - Spectral Sensitization Public Domain eBooks
 - Spectral Sensitization eBook Subscription Services
 - Spectral Sensitization Budget-Friendly Options
- 6. Navigating Spectral Sensitization eBook Formats
 - ePub, PDF, MOBI, and More
 - Spectral Sensitization Compatibility with Devices
 - Spectral Sensitization Enhanced eBook Features
- 7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Spectral Sensitization
 - Highlighting and Note-Taking Spectral Sensitization
 - Interactive Elements Spectral Sensitization
- 8. Staying Engaged with Spectral Sensitization
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Spectral Sensitization
- 9. Balancing eBooks and Physical Books Spectral Sensitization
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Spectral Sensitization
- 10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
- 11. Cultivating a Reading Routine Spectral Sensitization
 - Setting Reading Goals Spectral Sensitization
 - Carving Out Dedicated Reading Time
- 12. Sourcing Reliable Information of Spectral Sensitization
 - Fact-Checking eBook Content of Spectral Sensitization

- Distinguishing Credible Sources
- 13. Promoting Lifelong Learning
 - Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
- 14. Embracing eBook Trends
 - Integration of Multimedia Elements
 - Interactive and Gamified eBooks

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