

NANOSCALE FUNCTIONAL CERAMICS

Into the World of Photocatalyst

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1 Background: Recalling the Basic Concepts of Matter

1.1 Matter

Everything around us – from a grain of sand to a star – is called matter. In scientific words, anything which occupies space and has mass is matter. All matter in natural world is composed of one or more of the 118 fundamental substances which are known as Elements. An element is distinguished from all other matter by the fact that it cannot be created or broken down by any ordinary chemical means. But in nature, elements rarely occur alone. Instead they combine to form Compound which is composed of two or more elements joined by chemical bonds. In 1803, an English Mathematician named John Dalton said that matter consists of individual discrete particles. These tiny particle of an element were named as Atom, derived from the Greek word which means “Individual”. It is the smallest quantity that retains the unique properties of any pure element. For example, an atom of Hydrogen is a unit of Hydrogen, the smallest amount of Hydrogen that can exist.

Atoms are made of smaller subatomic particles, three of which are important – **Electron**, **Proton** and **Neutron**. (*In case you are wondering why it’s written as “three of which”, further details have been given in references for curious minds*). The number of positively charged protons and non – charged neutrons give mass to the atom and number of each in the nucleus of the atom determine the element. The negatively charged electrons spin around the nucleus at close to the speed of light and they are equal the number of protons. An electron has about 1/2000th the mass of proton or neutron. Rutherford and Bohr first helped us to visualize the structure of atom. Later, we have transferred our understanding from planetary model to electron cloud model. It is obvious that at the speed close to the light, the presence of electron cannot be identified at any specific point as showed in planetary model. This phenomena is known as “Heisenberg’s Uncertainty Principle”.

It can be understood from a simple example. When a ceiling fan rotates at a moderately high speed, we cannot see its wings though we know how many of wings it has. And while rotating, if we place any object in any place between the wings, it will be hit by the wings. It gives us the basic concept why rotation of electron is considered as a cloud and why its presence can be anywhere in the cloud.

Schrodinger mathematically explained the wave nature of matter and developed a wave equation to understand the electron’s behavior in the atom. The solution of the differential equation

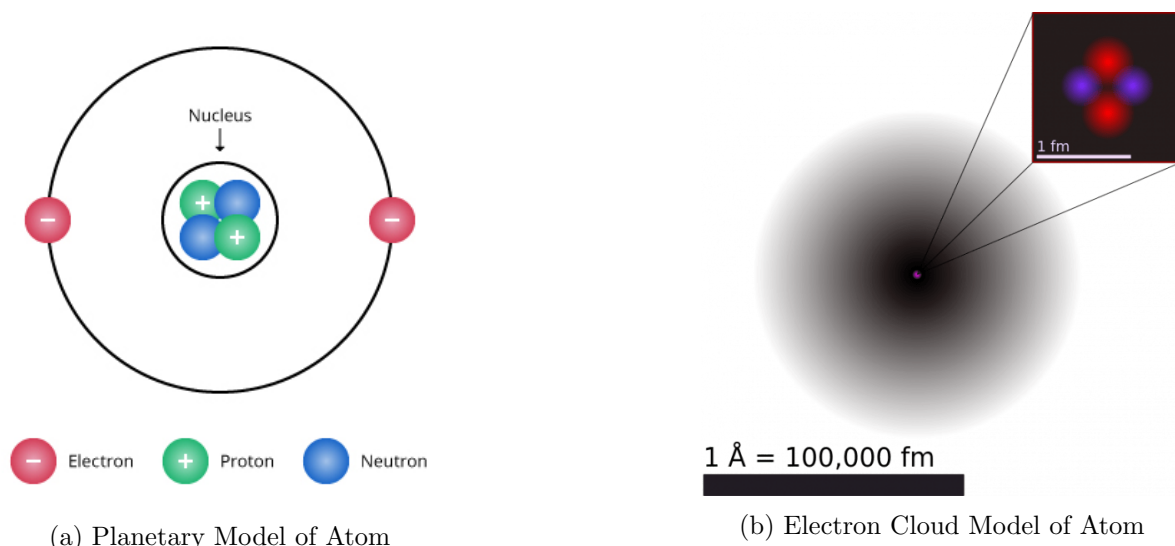


Figure 1: Schematic View of Atomic Structure

is known as wave function. Thus a concept of energy levels in the atom has been developed from the Bohr's atomic model. Schrodinger's derived wave function required three quantum numbers to explain Hydrogen or any multielectron atomic structure. These are designated by –

$$n = 1, 2, 3, \dots$$

$$l = 0, 1, 2, 3, \dots, (n - 1)$$

$$m_l = 0, \pm 1, \pm 2, \pm 3, \dots, \pm l$$

But from Pauli's Exclusion principle we came to know that no two electrons may occupy the same quantum state. So to specify a wave function completely, a fourth quantum number was introduced, the spin quantum number $m_s = +\frac{1}{2}$ or $-\frac{1}{2}$. **Food for thoughts**¹

1.2 Energy Band Theory

So far we have tried to explain the structure for a single atom. We have said that protons and neutrons remain in the nucleus, moving electrons form electron cloud and possibility of the presence of an electron can be identified in terms of energy states. But compounds are made up of two or more atoms. When two atoms come closer, their energy state of outer shell electrons are affected by neighboring atoms. The energy states of outer shell electrons become considerably changed and results in a band of closely separated energy states instead of widely separated energy states of an isolated atom. Most metals and semiconductors are crystalline in structure. In crystalline structure, a space array of atoms or molecules (specifically ions) is built up by the regular repetition in three dimensions of some fundamental structural unit (such as cubic, tetragonal, hexagonal etc.)

Let us consider a crystal consisting of N atoms of Si (14), $1s^2 2s^2 2p^6 3s^2 3p^2$ and also imagine that the distance between atoms can be varied according to our wish. When this distance is such bigger that no interaction between neighboring atoms take place, the energy states will coincide with those of the isolated Si atom. The outer shell of Si contains two electrons in s orbital and two electrons in p orbital.

¹ Do you know that we all are made of stars? Search in Google "Origin of Pure Elements". For better understanding of electronic behavior in atom, Khan Academy's videos can be watched. **Click Here**

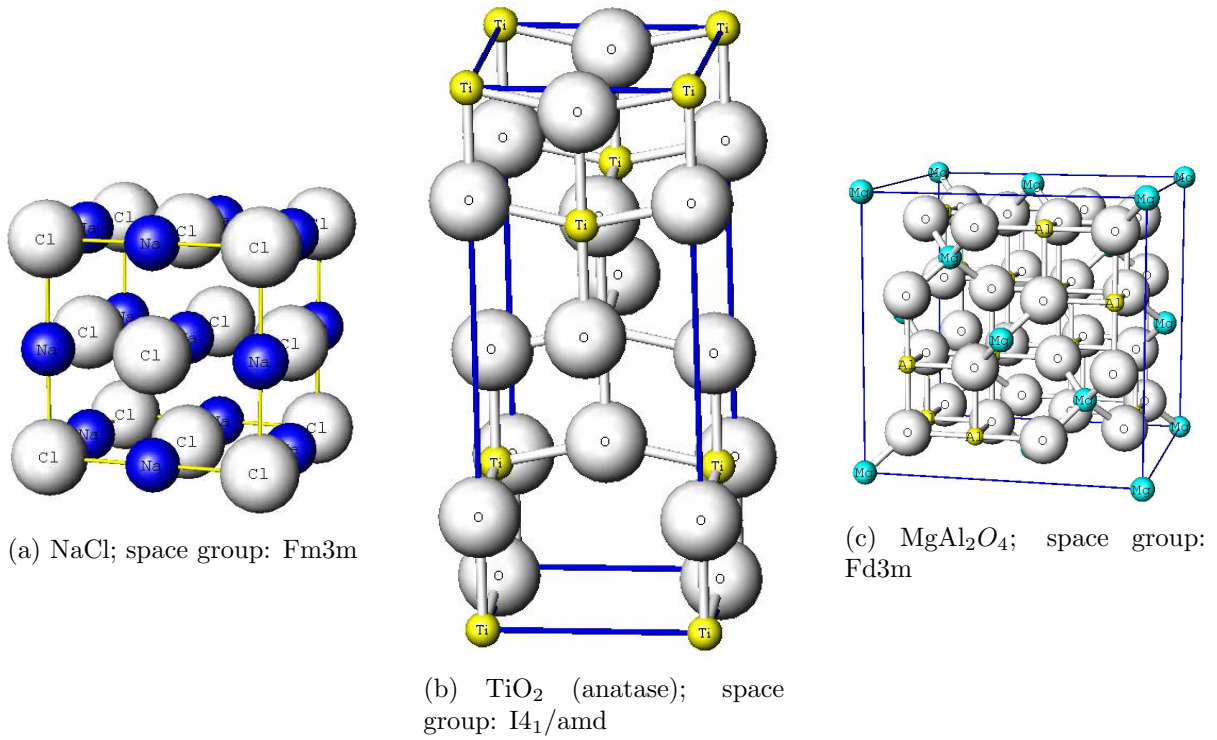


Figure 2: Crystal Structures of Different Symmetry

If we decrease the interatomic distance of our imaginary crystal, the energy states will overlap with each other. Because decreasing lattice parameters means atoms come close to each other. The atomic wave functions overlap and the crystal starts to behave like an electronic system which must obey the Pauli's Exclusion Principle. Hence the $2N$ s and $6N$ p energy states are generated. The separation between energy state is small but since N is very large. So the energy gap between the states may become several electron volts. This orientation of energy states is known as "energy band" and they are separated by a gap names as "forbidden zone". Up to a certain interatomic distance, the $2N$ s state is filled with $2N$ electrons and $6N$ p states are filled with $2N$ electron (Fig 3 a). With decrease in the lattice parameter, this forbidden gap decreases. Below critical point, these energy bands ($2N$ s and $6N$ p) merge with each other and form a total $8N$ energy bands. Half of which are occupied the $4N$ electrons and half remains empty. These $4N$ electrons then no longer belong to s or p orbitals rather they belong to the crystal as a whole. The bands occupied the electrons are known as "valence band" and the empty higher bands are known as "conduction band".

1.3 Metals, Semiconductors and Insulators

A nice approach of understanding the energy band theory is understanding the difference between metals, semiconductors and insulators. In metals, there is no band gap since two bands overlap. In semiconductor there is a moderate bandgap and in insulators the band gap is so large that at ordinary temperature no electron can reach the conduction band. The introduction of temperature concept is simple as we know that temperature represents the thermal energy (kT). In Fig 4, a new term named as "Fermi energy" appeared. The Fermi energy (E_F) is the highest energy state of electrons at $T = 0$ K. The Fermi function $f(E)$ gives the probability that a given available electron energy state will be occupied at a given temperature. It came

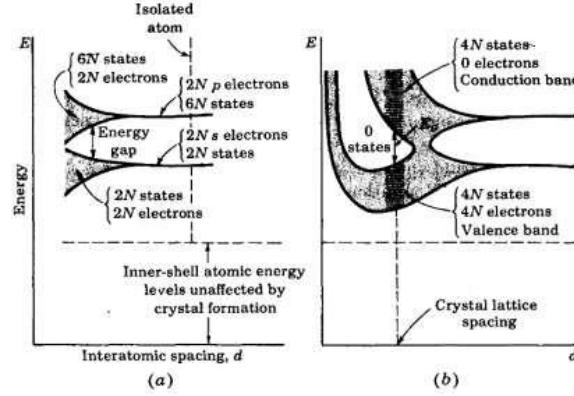


Figure 3: Formation of energy bands with varying the interatomic distance in crystal

from Fermi – Dirac Statistics.

$$f(E) = \frac{1}{e^{\frac{(E-E_F)}{kT}} + 1} \quad 2$$

1.4 Ceramics as Semiconductors

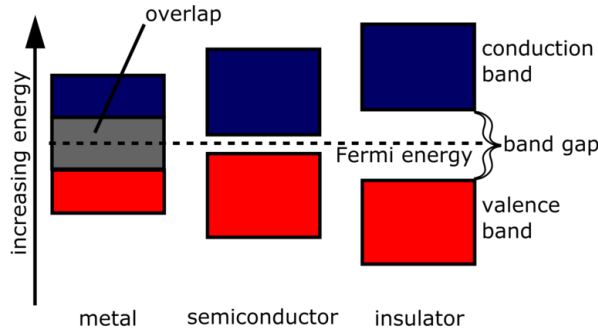


Figure 4: Energy band gap in metal, semiconductor and insulator

The most fascinating thing about semiconductor is its electron – hole recombination process. As we have seen that there is no bandgap in metal, there is an abundance of free electron which makes the recombination so fast that the presence of hole seems absent. When an electron jumps from valence band to conduction band, it leaves an empty place in valence band which is known as hole. From the definition it is clear that hole is positively charged. In quantum mechanics, hole is classified as “quasi – particles”. Just like the Fermi Sea of electrons, there is also a Dirac Sea vice versa. But it sounds like a strange idea for current conduction phenomena since the conduction in semiconductor is quite negligible compared to metal. But, this small amount of current conduction can be controlled and manipulated with the beautiful laws of physics. When a hole generates in valence band, the hole remains always hungry for the lost electron. The concept can be compared to the bank loan system. We lend an electron from semiconductor for any purpose and returns it after the purpose is done. The time between the electron – hole recombination is important for the purpose of any semiconductor.

2 Essence of Nanotechnology

A very common misconception about nanotechnology is nanoscale objects are high-tech things which can only be found in a mad scientist's laboratory. No! Nanoscale objects have been all around but we could not understand their existence. Chemists have dealt with naturally occurring nanoparticles all along. Think molecules or viruses. Toxicologists have dealt with nanoparticles that are the result of modern human life such as carbon particles in combustion engine exhaust. Without being aware of it, tire manufacturers used nanoparticles - carbon black - to improve the performance of tires as early as the 1920s. Artists used gold nanoparticles to achieve the bright red color in church windows (gold particles in nanometer size are red, not golden). In late 1980, the invention of STM (scanning tunneling microscopy) and AFM (atomic force microscopy) have given scientists new tools to understand and take advantage of phenomena that occur naturally when matter is organized at the nanoscale. With new tools came new concepts and it turned out that the mechanical rules that govern the nano world are quite different from our everyday macro world experience. These phenomena are based on "quantum effects" and other simple physical effects such as expanded surface area.

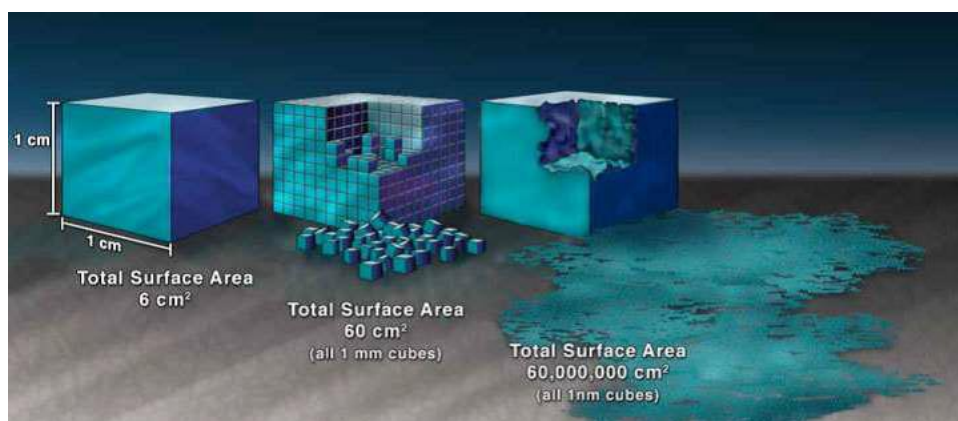


Figure 5: Illustration demonstrating the effect of the increased surface area provided by nanostructured materials

In addition, the fact that a majority of biological processes occur at the nanoscale gives scientists models and templates to imagine and construct new processes that can enhance their work in medicine, imaging, computing, printing, chemical catalysis, materials synthesis, and many other fields. Nanotechnology is not simply working at ever smaller dimensions; rather, working at the nanoscale enables scientists to utilize the unique physical, chemical, mechanical, and optical properties of materials that naturally occur at that scale.

2.1 Quantum Confinement Effect

When particle sizes of solid matter in the visible scale are compared to what can be seen in a regular optical microscope, there is little difference in the properties of the particles. But when particles are created with dimensions of about 1–100 nanometers (where the particles can be “seen” only with powerful specialized microscopes), the materials’ properties change significantly from those at larger scales. This is the size scale where so-called quantum effects rule the behavior and properties of particles. Quantum confinement effect is essentially due to changes in the atomic structure as a result of direct influence of ultra-small length scale on the energy band structure. The length scale corresponds to the regime of quantum confinement ranges from 1 to 25 nm for typical semiconductor groups of IV, III-V and II-VI. In which the

spatial extent of the electronic wave function is comparable with the particle size. As a result of these "geometrical" constraints, electrons "feel" the presence of the particle boundaries and respond to changes in particle size by adjusting their energy. This phenomenon is known as the quantum-size effect or simply quantum effect. A fascinating and powerful result of the quantum effects of the nanoscale is the concept of "tunability" of properties. That is when particle size is made to be nanoscale, properties such as melting point, fluorescence, electrical conductivity, magnetic permeability, and chemical reactivity change as a function of the size of the particle. But size is not the only one which controls the properties. As nanotechnology moves towards applications in electronics and medicine, it's becoming clear that shape plays an equally important role in controlling nanoparticle properties.

3 Basic Concepts of Photocatalyst

3.1 Photocatalyst and Photocatalytic Activity

During any chemical reactions, breaking and rebuilding of chemical bonds happen between atoms. Sometimes the partnerships may easily break and rebuild new bonds with new atoms. Sometimes the molecules want to remain stable and resist to build new bonds. For this case, the need of a mutual friend becomes necessary who will come and break the ice between molecules but does not take part in the event. These unsung heroes are known as Catalysts.

Catalysts are the substances that reduce the activation energy and speed up the chemical reaction. Photocatalysts are some special type of catalyst who need light as well as photon energy to become activated as a catalyst for any chemical reaction.

In late 1970, study on photocatalysts became accelerated just after the famous work of "Honda-Fujishima effect" on photo electrochemical water splitting using a single-crystal TiO₂ electrode [1]. In the field of catalysis, "catalytic activity" has been used to show a property or performance of a catalyst, since an "active site" (Fig 6) on a catalyst accounts for the catalytic reaction. The reaction rate per active site can be estimated and should be "catalytic activity". Sometimes the term "turnover frequency" as well as the number of turnovers per unit time of reaction is used to show how many times one active site produces a reaction product(s) within unit time. On the other hand, there are no such active sites in photocatalysts and reaction rate strongly depends on various factors such as the intensity of irradiated light which initiates a photocatalytic reaction.

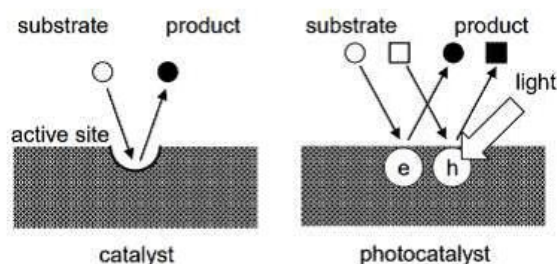


Figure 6: Difference in concepts of catalytic and photocatalytic reactions: A catalyst contains active sites of which a substrate is converted into a product, while no active sites are present on a photocatalyst

In kinetic analysis of general chemical reactions, a rate constant is estimated and compared. Considering that photo-excited electrons (e^-) and positive holes (h^+) induce a redox reaction, it may be possible to estimate the rate constant of these active species. Since e^- and h^+ recombine

with each other ("Mutual recombination"), the overall photocatalytic reaction rate depends also on this recombination rate. Assuming that k (redox) and k (recombination) are rate constants of reactions by e^- and h^+ and their recombination, respectively, i.e., the simplest kinetic model, the ratio k (redox) / k (recombination) should be a measure of intrinsic photocatalytic activity. However, unfortunately, we have no way to estimate k (recombination), since the recombination does not produce any chemical species to be detected for estimation of its rate. For that reason no true universal correlation has been reported yet between physical or structural properties and photocatalytic reaction rate. In layman's terms, no physics have been developed yet for any desired photocatalysts which acts as the driving force for many researchers all over the world.

3.2 Quest for a Photocatalyst

As days keep passing, we are running out of oils and other nonrenewable fuels. If we see around, we will notice that two mostly seen abundant things are sunlight and water. We all know that 2 mole of Hydrogen and 1 mole of Oxygen make up water. So, if we just split water and produce Hydrogen gas, we will be able to use this gas as clean burning fuel which does not produce greenhouse gases.

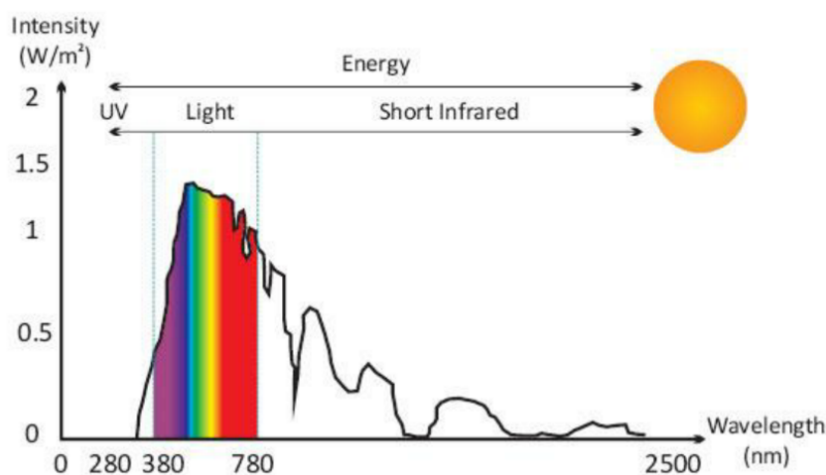


Figure 7: Solar Energy Spectra

But, splitting water into Hydrogen and Oxygen is not an easy process rather it is highly endothermic process. The splitting of water requires large amount of energy which makes this process an expensive one except it can be mitigated by the abundance of solar energy. So, if any photocatalyst can be produced which will capture the sunlight and make the water splitting naturally possible. Though no such magical photocatalyst has been found yet, the quest is still unsatisfied to the researchers. Apart from this example of water splitting, there are more fields waiting eagerly for the desired characterized photocatalysts such as degradation of pollutants, photothermal activities in cancer treatment and antibacterial approaches.

We know that wavelength range of visible light is (380 nm – 750 nm). According to $E = h\nu$, the lowest (for 380 nm) and highest energy (for 750 nm) can be achieved are 3.27 eV and 1.66 eV respectively. This provided energy must be equal or greater than the band gap energy (E_g) of the photocatalyst to free the electron from its valence band to conduction band. The photocatalyst which has the bandgap lower than 1.66 eV can surely utilize the full range of visible light. It should be mentioned that only decreasing the bandgap does not guarantee of enhanced photocatalytic activity. Size and shape morphology play an important role along with

the controlled bandgap which is already mentioned earlier in this study. We will try to give a short overview of current researches of some well-known photocatalysts later at the end of the study.

3.3 Mechanism: Photocatalytic Activity

The general understanding of the mechanism of Photocatalysis is that photoabsorption of a semiconducting material occurs to excite electrons from the valence band (VB) to the conduction band (CB) leaving positive holes in the VB, i.e., electron-hole pair (e^- and h^+) generation (Fig. 8). An important point is that photoabsorption and (e^- and h^+) generation are strongly linked. This inter band (band-to-band) excitation is often illustrated by three bands, CB, forbidden band (band gap) and VB, in which an electron moves vertically from the VB to CB, i.e., no spatial change in the position of an electron, though misunderstanding may happen in saying that an electron migrates from the VB to CB spatially.³

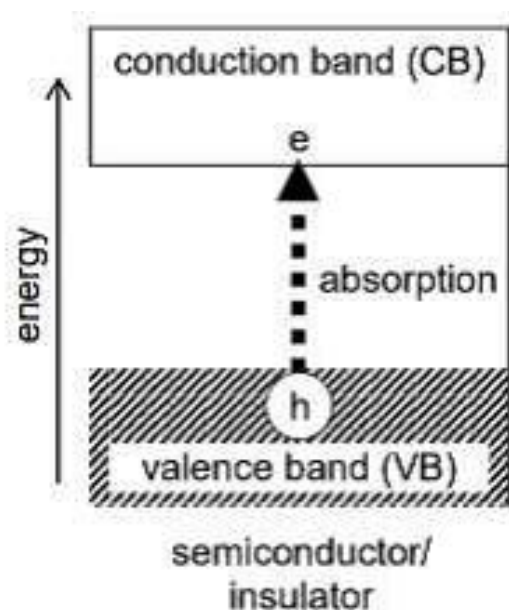


Figure 8: Photoabsorption by transition of electrons in the valence band (VB) to the conduction band (CB) in a semiconductor

The above-mentioned interpretation seems to be a little strange considering the meaning of band structure and band-to-band transition, in which electrons are not localized and therefore electrons and positive holes can migrate within a crystal; an unlocalized excitation state may be described as "photoexcited crystal" without showing localized e^- and h^+ . Do e^- and h^+ migrate in the CB and VB, respectively, after photoabsorption, i.e., photoexcitation? When we illustrate the electronic structure of a molecule, lines are drawn to show the electronic state (Fig. 8); the length of these lines does not mean spatial distribution of electrons in those states. This should also be the case for semiconducting (or insulating) materials, and band-to-band transition just means that an electron in the VB is excited to the CB without clarifying the location of e^- and h^+ . A possible interpretation of a better understanding for e^- and h^+ location is that there are sites trapping e^- or h^+ in the crystal lattice and that e^- and h^+ are trapped by these sites "immediately" after the band-to-band transition, i.e., photoabsorption. Location of e^- and h^+ in the initial stage of Photocatalysis as well as the rate should be controlled by

³ To gain better understanding of the electronic transfer, clear concept of brillouin zone is necessary. Details: Semiconductor Physics and Basics (Chapter III: Introduction to Quantum Mechanics) by Donald A. Neaman.

the density and spatial distribution of these traps in a photocatalyst. However, there is little information on the density and spatial distribution of traps, since the structure of traps cannot be fully understood. The possible surface traps may be reaction sites, but not "active sites".

Fermi level is a kind of measure of equilibrium electrochemical potential of a solid material. It is thought that Fermi level is located just below the CB bottom and above the VB top for n-type and p-type semiconducting materials respectively. Most metal oxides are categorized as n-type semiconductors with Fermi levels more cathodic (higher) than the standard electrode potential of an electrolyte in contact with the metal oxide, and thereby electrons in donor levels slightly below the CB are injected into the electrolyte to form a space charge (depletion) layer with an electric field, i.e., Schottky barrier. In the 1980's, it was thought that this inner electric field separates e^- and h^+ effectively as well as e^- and h^+ migrate to the bulk and surfaces of semiconductor electrodes and particles, but it seems that this is not the case for untreated photocatalyst particles because of the expected large thickness of this layer due to very low density of donor levels in ordinary photocatalyst particles [2]. In simple words, Photocatalytic activity is nothing but a redox reaction which generates superoxide anions and hydroxyl radicals which are known as reactive oxygen species (ROS). They are short lived and highly capable of breaking any chemical bonds.



ROS (Reactive Oxygen Species) is the key word in Photocatalysis. They create oxidative stress under heat exposures. ROS are able to damage cell membranes, DNA, RNA and currently regarded as most promising hopes for fighting against cancer and microbial resistance of bacteria. Some important ROS are: peroxides, superoxide, hydroxyl radical and singlet oxygen. Fluorine is the most powerful oxidizing element (relative oxidizing power 2.23).

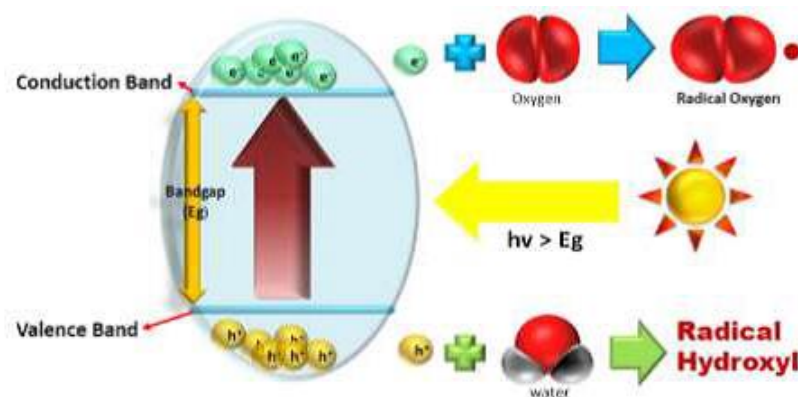


Figure 9: Mechanism of Photocatalytic and Photothermal Activity

It is clear from the Fig 6 that energy bandgap E_g plays the vital role for the activation of

| Relative Power of Chemical Oxidants | | | | | |
|-------------------------------------|-----------------------------|--|------------------|-----------------------------|--|
| Compound | Oxidation Potential (Volts) | Relative Oxidizing Power (Cl ₂ = 1.0) | Compound | Oxidation Potential (Volts) | Relative Oxidizing Power (Cl ₂ = 1.0) |
| Hydroxyl radical | 2.8 | 2.1 | Chlorine Dioxide | 1.5 | 1.1 |
| Sulphate radical | 2.6 | 1.9 | Chlorine | 1.4 | 1.0 |
| Ozone | 2.1 | 1.5 | Oxygen | 1.2 | 0.90 |
| Hydrogen Peroxide | 1.8 | 1.3 | Bromine | 1.1 | 0.80 |
| Permanganate | 1.7 | 1.2 | Iodine | 0.76 | 0.54 |

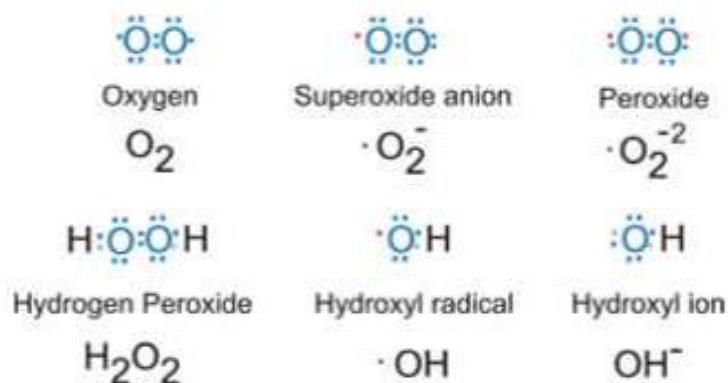


Figure 10: Reactive Oxygen Species

photocatalyst. So the desired photocatalysts should have a minimum energy bandgap and high surface area to engage itself quickly and efficiently in the photocatalytic activity.

3.4 Energy Conversion in Photocatalytic Activity: Water Splitting

Photocatalytic and photo electrochemical mechanism have made researchers hopeful for naturally occurring water splitting to generate hydrogen (H_2) as an ideal and green fuel (no carbon emission) and many researchers are trying to establish a highly efficient system for water splitting under solar radiation. Since this reaction requires input of energy due to its positive Gibbs energy, energy of light is used. The efficiency of conversion of light energy to chemical energy thus becomes important. It should be noted that there are at least two kinds of methods for calculation of the efficiency: number (molar amount) - based and energy - based methods. The former is the same as "quantum efficiency", which is calculated as a number ratio of product(s) and photons absorbed by (quantum efficiency) or incident on the reaction system (apparent quantum efficiency; ϕ in Fig. 11).

For discussion of energy conversion, the energy-based calculation should be used. Since the energy of H_2 (and O_2) shown in the difference in electrochemical potential, i.e., electromotive force (emf), is 1.23 eV, energy conversion efficiency is 100% when light of 1.23 eV energy (1000 nm wavelength) is absorbed completely by a photocatalyst and all liberated e^- and h^+ are used for water cleavage. The most significant point of Photocatalysis and photo electrochemical reaction is that even if light of energy much greater than the band gap of semiconducting materials as a photocatalyst or photoelectrode is used, potential of e^- and h^+ is fixed at the position of the CB bottom and VB top, respectively. Therefore, the energy conversion efficiency is halved when 2.46-eV light (504 nm) is used with constant apparent quantum efficiency (Fig. 11 a).

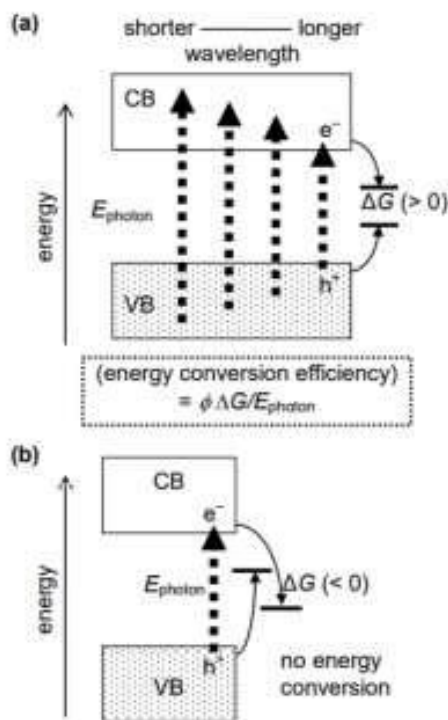


Figure 11: (a) Light – chemical energy conversion efficiency of photocatalytic reaction calculated as an integral of a product of apparent quantum efficiency (ϕ) and Gibbs energy change (storage) divided by photon energy (E_{photon}) as a function of wavelength. (b) In the case where Gibbs energy change is negative, energy conversion efficiency cannot be defined, or is defined to be zero.

An appropriate method for calculation of energy conversion efficiency for photocatalysts is still missing to the best knowledge of this author.

3.5 Mutual Recombination: The Reduction in Quantum Efficiency

Recombination of a photoexcited electron (e^-) and a positive hole (h^+) occurs to some degree in photocatalysts, and this reduces quantum efficiency, i.e., efficiency of $e^- - h^+$ used in chemical reaction(s), and overall photocatalytic reaction rate. Since recombination does not produce any chemicals, it is not easy to estimate the rate of recombination directly. One possible way for estimating recombination rate is to subtract the overall rate of chemical reaction by e^- and h^+ from the rate of photoabsorption, but the obtained data cannot give any other information. Kinetics of e^- and h^+ recombination may depend on its fashion. If one electron is excited and this is recombined with h^+ , the recombination rate obeys the first-order rate law, while if multiple e^- and h^+ appear at the same time within a photocatalyst particle, the rate obeys the second-order rate law [2]. However, estimation of any absolute rate constant of mutual recombination occurring during photocatalytic reaction under ordinary photo-irradiation systems has been reported yet.

3.6 Antibacterial Activity: Mechanism

Our body has its own anti pathogen mechanism (white blood cells) but with the evolution of bacteria, we need some external help along with the white blood cell to fight against it.

Hence, antibiotics were discovered and have been developed since the invention of penicillin. Antibiotic's mechanisms are:

- Cell wall synthesis (damage the cell membrane)
- Translated Machinery (to stop the translation process of bacteria)
- DNA replication machinery (to stop DNA replication and to prevent its evolution)

The alarming thing is bacteria can resist these all 3 mechanisms and they seem to learn from the antibiotics' activity so that they can continue their evolution in future ignoring that previously applied antibiotics. Already a bacteria (NDM – 1) has been found which has super resistance against all available antibiotics!

There are two reasons have been found for which a bacteria becomes super drug resistant. They are:

- Intrinsic resistance: Spontaneous mutation takes place in the existing genes. This is less important/serious problem.
- Extrinsic resistance: Collecting resistant genes from other cells. Severe reason.

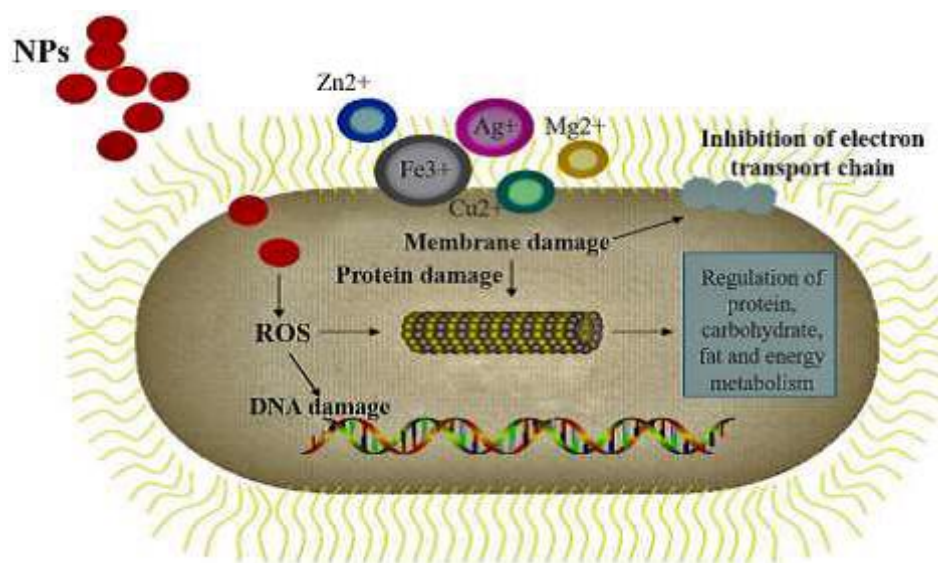


Figure 12: Mechanism of NP action in bacteria cell

Nanoparticles have been found as the desired alternatives of the antibiotics. The main advantage of NPs over antibiotics is NPs don't have to penetrate the bacterial cell to destroy it. Bacteria cannot resist NPs' antibacterial activity. NP's antibacterial mechanisms are:

- Generation of reactive oxygen species (ROS) and oxidative stress induction
- Metal ion release
- Non – oxidative mechanisms

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