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LITERATURE REVIEW ON ORGANIC MATERIALS FOR THIRD HARMONIC OPTICAL AND PHOTONIC APPLICATIONS

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Abstract:

The third harmonic optical applications such as optical phase conjugation, image processing, optical switching, and optical limiting in Photonics requires efficient nonlinear optical materials to be used with low power laser beams. During the last three decades, organic molecules have attracted the attention of many researchers due to their high nonlinear optical susceptibility and the possibility of tailoring their properties suitable to be used to protect optical detection components and devices such as human eyes and optical sensors, by controlling the output energy on the image plane below the desired level. Extensive studies have been performed and reported for the case of single crystals of organic molecules, organic molecules in liquid solutions, and organics and organometallics doped in various solid substances. One of the major benefits of organic optical materials is that they are usually much less costly and easier to process in device form than their inorganic materials. In this paper, an extensive literature survey on nonlinear optical materials is depicted. This includes review of organic nonlinear material research, materials for reverse saturation absorption, molecules for two-photon and multi-photon absorption, experimental techniques for nonlinear refraction, nonlinear absorption, optical limiting, and optical phase conjugation. Finally, the review includes the materials used in photonic devices, materials used for optical phase conjugation like nonresonant media and resonant media including absorbing liquid and solid materials, lasing gain media, metal vapors, and photorefractive materials.

Index Terms: Organic Materials for Nonlinear Optics, Organic Dyes, Dye-Doped Polymers & Nonlinear Materials Research

1. Introduction:

Basically, all materials exhibit nonlinear optical phenomena. This includes all forms of matter – gases, liquids, and solids. The power of the optical fields required to observe these effects varies over many orders of magnitude, depending on the detailed nature of the electronic structure of the atomic and molecular arrangements of the medium, their dynamical behaviour, as well as the symmetry and details of their geometrical arrangement in the medium. The important nonlinear optical materials from the device point of view are generally in solid formats and must meet a wide variety of ancillary material requirements for practical use. In general, they require extraordinary stability with respect to ambient conditions and high intensity light sources. They will have to meet many processing requirements for device pattern, and integration with additional dissimilar materials. Some of the affecting mechanisms studied for the optical limiting effect in materials are reverse saturation absorption, multi-photon absorption, photo-refractivity (due to beam fanning), linear and nonlinear scattering, self-focusing and defocusing, free carrier absorption, intermolecular charge transfer absorption, photo-chromics, and liquid crystal fibre arrays.

2. Literature Review:

During last five decades research on third order optical materials for vatious applications in Photonics are active and several conferences have been held on the topics like optical power limiting, Optical switching, Optical phase conjugation etc. and their proceedings have been published [1-2]. There have been reviewson optical limiting studies in various materials by Tutt and Boggess [3], Perry [4] and Van Strylandet. al. [5]. The various requirements for an effective optical power limiter have been outlined by Justus et al [6], Miles [7], and Miller et. al [8], as given below :

- ✓ A reduction of laser fluence (energy per area) by a factor of 10^4 to 10^5. This is a big order to fill. ANSI standard prescribes a maximum fluence of 0.5 $\mu J/cm^2$ for laser pulses shorter than 17μs. This reduction in fluence has not been demonstrated with any material till date.
- ✓ For eye protection, we need a broad spectral range of response that is we need to protect from 400nm to 700 nm device at low light intensity levels. High transmission of 50% to 70%, for any limiting, is also required.
- ✓ A fast temporal response and rapid recovery time is needed.
- ✓ High threshold for optical damage and recovery is needed.
- ✓ A solid based device with a large index change is much preferred over any liquid based device.

These are difficult conditions to be met, but this survey is limited to visible range. In general, the nonlinear absorption is associated with the decrease of the intensity, I, of high beam travelling through a nonlinear medium in the z-direction, which can be described by the following equation:

$$\delta I/\delta z = -\alpha I - \beta I^2 - \gamma I^3 - \ldots\ldots\ldots \quad (1)$$

Where α, β, and γ are the linear absorption coefficient, two-photon absorption coefficient, and three-photon absorption coefficient respectively. Higher terms in equation (2.1) can also contribute through higher order photon absorption, but usually are insignificant.

Reverse Saturation Absorption:

In the late 1960's Giuliano and Hess [9], were looking for better saturation absorbers for Q switching a laser. Under high intensity the ground state to first excited state transition is saturated and no longer absorbs, opening a way for high pulse of radiation to be emitted from the laser cavity. Although they found many compounds for saturation absorption, they found at least one compound, platinum dithizonate, which exhibited both positive and negative saturation effects [10]. It is also found that the optical transmission as a function of incident power density goes through a maximum and decreases at higher power densities. The effect is very pronounced and should be useful in laser applications i.e., in passive power limiting devices. Historically this is one of the first references to Reverse Saturation Absorption (RSA).

In 1935 Jablonski [11] was the first researcher to draw a schematic energy diagram for dye molecules as shown in Figure 1. For good RSA behavior, the transition probability for the first excited state to high states must be significantly higher than the absorption probability from the ground singlet state to the first excited singlet state. This ratio (σ_2/σ_1) of the two absorption probabilities should be greater than 30 for good RSA process. Approximate values for the relaxation of the first excited state to the ground state is of the order of 10 ns, while intersystem crossing to the triplet state can be slower at least one order of magnitude. On the other hand, relaxation times from the second excited state to the first excited state can be in the picosecond range, a very much faster process. For an efficient RSA process both the excited state absorption in the singlet manifold and the absorption from the lowest triplet state are required. Usually, if $\sigma_2 >> \sigma_1$ the RSA process is dominated by a multiphoton absorption mechanism. Recently, during last four years many papers in optical power limiting have been published by many researchers [12-18].

Two and Multi Photon Absorption:

Two photon absorption can be another effective way to achieve optical limiting. The possibility of a simultaneous absorption of two photons is predicted theoretically in early thirties of twentieth century by Maria Goppert [19]. According to Maria Goppert, the excited levels of a centrosymmetric molecule can be classified into "gerade" and "ungerade" levels differing in their total angular momentum quantum number by an integral odd number. Because photons bear a spin of 1, the light induce transitions between states of opposite symmetry (gerade to ungerade or opposite) are allowed. However, with any even number of photons it is possible to excite electrons between states of the same symmetry. The transition involving the lowest number of photons is a two photon transition, which is a simultaneous absorption of two photons.

The first transition is to a virtual state, followed by excitation to a final state with symmetry like that of ground state (Fig. 2). The states accessible by absorption of two (or any even number) photons are called two photon states. What is interesting here is that the transitions to a two photon state are forbidden for one (or any odd number of photons) photon. Consequently, the molecule is transparent for photon energies corresponding to the transition energy between fundamental and the two photon state. In non centrosymmetric molecules, such as charge transfer molecules, due to the presence of a permanent electric field the selection rules are relaxed and two photon transitions are possible between all states. Insuch case the molecule absorbs light for photon energies corresponding to the transition energy between the fundamental and the two photon state. However, it corresponds to half of the two photon excitation transition energy. Thus, these materials are similar to centrosymmetric materials, that is, the molecules are mainly transparent when excited by two photons [20-23].

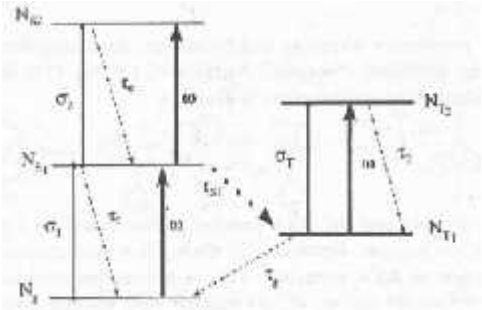

Figure 1: Schematic representation of RSA Process [19].

3. Organic Nonlinear Optical Materials Research:

The need for nonlinear optical materials and devices that can be used with low intensity continuous wave laser light beams for applications such as optical limiting, optical switching, optical phase conjugation,

image processing, and optical memory storage have increasingly become important [24-25]. Recently, organic molecules have attracted much attention due to their large nonlinear optical susceptibility and the possibility of tailoring their properties which allow these materials to be used in passive optical limiters to protect optical detection elements such as human eyes and optical sensors. Such optical limiters can be used to control the fluency of high intensity laser beam on the image plane below the desired level. Extensive research work have been done and reported for the case of single crystals of organic molecules [26-27], organic molecules in liquid solutions [28-31], organics, and organometallics doped in various solids including glasses and polymers [32-34]. One of the big advantages of organic optical materials is that they are easier to fabricate in the form of required structure and also less expensive than their inorganic counterpart.

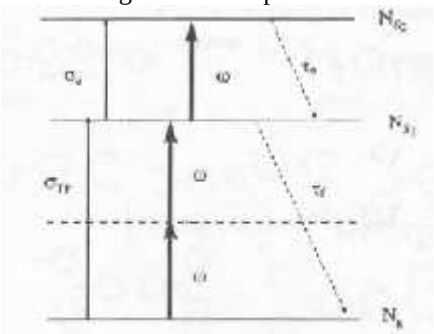

Figure 2: Schematic representation of a two and three photon absorption process [19].

An optical limiter device used to limit high intensity laser beam can be fabricated using organic materials works on various nonlinear mechanisms such as Reverse Saturation Absorption (RSA), Two-Photon Absorption (TPA), Two-Photon Fluorescence (TPF), nonlinear refraction (NR), and nonlinear scattering (NS). Out of these, two-photon induced absorption or two-photon induced fluorescence possesses the advantages like high initial transmission for a weak input optical beam, instantaneous response to a change of input intensity of the optical beam and retention of optical quality of the input beam after transmitting through the nonlinear material medium. Apart from these, TPA process depends on both a spatial and temporal overlap of the incident photons and depends on a quadratic nonlinear property of the molecules of the material which depends on the incident light irradiance. The quadratic dependence of absorbed incident light intensity results in characteristics like high localized photo-excitation, inherent three dimensional spatial resolution into two-photon absorption, deeper penetration depths with near-IR light resulting higher transparency of materials at longer wavelengths, lower photodamage, and photobleaching. These advantages of two-photon absorption and two-photon fluorescence can be utilized not only for high-intensity protection for optical devices but also for stabilizing fast fluctuations of laser power or intensity [35].

Two-photon absorption cross-sections have been studied primarily by measuring the optical fluorescence resulting from the decay of a species excited by absorption of two photons. This technique had several drawbacks associated with spatial and temporal characteristics of the laser beams. Uncertainties in the spatial distribution of the light intensity can introduce large errors into the measurement through the cross section's dependence on I^2. This technique requires measurement of absolute intensities and an accurate knowledge of quantum yield for optical fluorescence. All these factors lead to large uncertainties in the measured two-photon cross section values. Several years ago Levenson, Bloembergen, and co-workers, [36-37] pointed out that a measurement of a liquid's third order susceptibility allows determining the two-photon absorption cross section for a given liquid. Later Hochstrasser et al. [38] demonstrated that the technique could be applied to gaseous molecules. The technique proposed by Hochstrasseret. al. involves the measurement of the third-order susceptibility χ^3 in two gasses, one of unknown χ^3. The measurement thus became a relative measurement with all attendant improvement in experimental reliability. In 1985, J. Burris et al.[39] employed a four-wave mixing method for determination of two-photon absorption cross section of diatomic molecules. The technique involves the measurement of third-order susceptibility, through observation of a two-photon-resonant four-wave signal and extraction of χ^3 and two-photon coefficient. Other techniques include nonlinear interferometry [40-41], degenerate four-wave mixing [42], nearly-degenerate three-wave mixing [43], ellipse rotation [44], and beam distortion measurements [45-46]. Nonlinear interferometry, four-wave mixing, and three-wave mixing techniques are optically sensitive techniques but require relatively complex instruments and experimental setup. The beam distortion techniques, on other hand, are relatively insensitive and require detailed analysis of light wave propagation through the medium. In 1989, Sheik-Bahaeet. al. from CREOL, University of Central Florida [47-48] demonstrated a new simple technique for measuring the value of n_2 and hence two-photon cross section called Z-scan technique. The principle of working of this technique is based on the spatial beam distortion by the nonlinear sample. This technique offers simplicity as well as very high sensitivity for measuring the nonlinear refractive index of the material.

4. Materials for Reverse Saturable Absorption:

The currently studied materials for RSA can be conveniently divided into three types: carbon related materials, aromatic ring structures commonly containing a metal ion complex at the center, and dyes. The carbon related materials are a subject of an intense study for optical power limiting. In particular carbon black in solution (inks) [49-50] and Nigrosin [51] (a black staining dye for biological systems) show excellent performances. But concerning nonlinear absorption funerenes (C_{60} and C_{70}) [52-54] belong to a class of better RSA materials. All these materials absorb over a broad visible wavelength range - a good attribute. The fullerenes, on the other hand, are frequently chemical1y modified with side groups to make them more soluble into a solvent or to be efficiently adsorbed onto a solid, like sol-gel. Recently charge transfer complexes are also under research for optical power limiting such as combinations of C_{60} compounds and zinc phthalocyanine [55]. The phthalocyanine [55-59], naththalocyanine [58] and porphyrin [60-64] systems are ring systems comprised of four aromatic ring systems joined together with either two hydrogen atom in the middle or a metal ion which forms a complex. Some of the metals are zinc, silicon, cadmium, lead, tin, gallium, indium, and germanium, to name a few. As mentioned above, the complexes with heavier metals seem to function more efficiently. Many other compounds both organic and inorganic materials also have saturation and reverse saturation absorption [65-72].

5. Molecules for Two and Multi Photon Absorption:

Two types of molecules were studied for two (and multi) photon absorption:

- ✓ Small molecules composed of a conjugated backbone (π) and electron donating (D-π-D), electron attracting (A-π-A) or mixed electron donating - electron attracting side groups (D-π-A) [73-74]: Small molecules usually exhibit a sufficiently large optical band, thus are transparent over a large part of the visible spectrum. However they are difficult to process and are used mainly in solution form. Theoretical calculations, as well as experimental results, indicate that A-π-A type molecules are the most efficient TP absorbers [75-76].
- ✓ Conjugated π electron polymers [77-82]: The main advantage of conjugated polymers is that some of them are soluble and can be processed into good optical quality films or slabs. Also often, due to the large electron-electron correlation the two-photon states lies below the one photon state. The main disadvantage is that they have usually a small optical gap which limits the applicability of these polymers for the optical power limiting in the visible spectral region.

Two photon absorption (TPA) based optical power limiting has been studied in organic dyes either in solution form or in solids. However, direct use of organic dyes for TPA or TPF has a disadvantage of intense light induced degradation or bleaching and aggregation at higher dye concentration. In order to overcome these drawbacks and for effective use of highly nonlinear dyes, they can be doped in polymer matrix. This may increase the concentration of absorptive or fluorescence centers as well as the opto-chemical andopto-physical stability.

6 Experimental Techniques:

6.1 Third Order Nonlinearity Study:

Direct Techniques: Clearly the simplest and most direct method is to measure the output fIuence as a function of input fluence. This give the reduction directly. In this single beam technique developed by Sheik Bahae and Van Strayland of University of Central Florida to measure the magnitude of nonlinear absorption as well as the sign and magnitude of nonlinear refraction and called Z-scan method. In Z-scan method, a laser beam propagating through a nonlinear medium will experience both amplitude and phase variations. If transmitted light is measured through an aperture placed in the far field with respect to focal region, the technique is called closed aperture Z-Scan. In this case, the transmitted light is sensitive to both nonlinear absorption and nonlinear refraction. On the other hand without an aperture, the mode of measurement is referred to as open aperture Z-Scan. Z-Scan methods yield the real and imaginary parts of nonlinear susceptibility $\chi^{(3)}$ respectively. Usually closed aperture Z-scan data are divided by open aperture data to cancel the effect of nonlinear absorption contained in the closed aperture measurements. The new graph called divided Z-scan contains information on nonlinear refraction alone [83-87]. The main drawback of both techniques is that they give only the global two (or multi) photon absorption coefficient without discriminating the origin. Although this is not important from the point of view of practical application of studied materials in optical power limiting devices. It does limit the understanding of the origin of the nonlinear absorption and the possibility of material optimization. Indeed, different physical effects may contribute to the variation of beam transmission through a nonlinear medium. Only time resolved techniques or use of ultrafast laser pulses could lead to a resolution.

Indirect Techniques: Pump-probe techniques have been used to measure the temporal characteristics [88]. The most useful is the Kerr ellipsometric technique which allows one to measure the dispersion of nonlinear refraction and absorption coefficients. Degenerate four wave mixing (DFWM) also gives the same information. The Kerr $\chi^{(3)}(-\mu;\mu,-\mu,\mu)$ susceptibility can be used to derive the β coefficient and subsequently the two photon absorption cross section. Finally, $\chi^{(3)}$can be measured by third harmonic generation (THG). The main advantage

of this technique is that it measures the fast, electronic part of real and imaginary part of $\chi^{(3)}$susceptibility. These may be the used to calculate the Kerr susceptibility $\chi^{(3)}(-\mu;\mu,-\mu,\mu)$ via multilevel quantum models. Single photon spectroscopic results are also useful to characterize potential materials.

6.2 Phase Conjugation Wave generation using Four-Wave Mixing:

In the area of optical phase conjugation (OPC) research, the majority of experimental studies have been carried out by using the degenerate four-wave mixing (DFWM) method. The reason for this is that the principle and visualization of that method are simple and clear, the required laser facilities are relatively inexpensive, and most importantly, a huge number and variety of materials can be used as nonlinear optical media for tests. It is seen that a considerable amount of such experiments have been focused on determining the nonlinearity of materials rather than studying the phase-conjugate properties of backward generated optical beams [89]. Fig. 3 shows two set of experimental configurations for studying phase conjugation wave via backward degenerate FWM. In these experimental configurations, the incident beam from a laser source is divided into three beams via appropriate mirrors and beam splitters. Two of them are used as pump beams and pass through a nonlinear medium in a counter-propagating way, while the third beam containing certain spatial information is incident upon the nonlinear medium with a crossing angle to one of the pump beams. Under these arrangements the backward phase conjugated wave can be measured in the direction opposite to that of the incident signal beam. To ensure a longer interaction length in the sample film, the angle between the signal beam and one of the pump beams should be relatively small. In practice, to achieve a higher local intensity the three incident beams are either weakly focused by a lens system or compressed with a reversed beam expander.

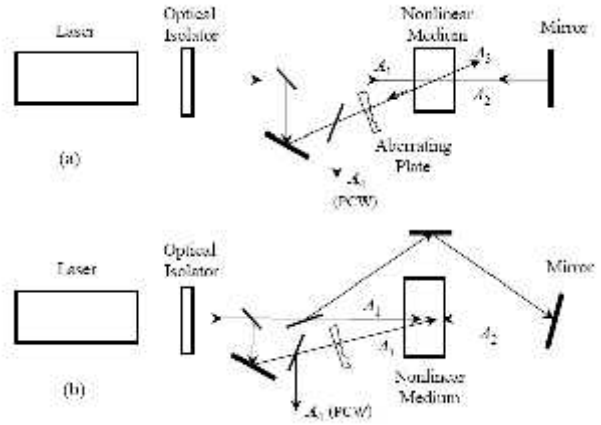

Figure 3: Two typical experimental setups for PCW generation via degenerate four-wave mixing [89]

The difference between these two layouts is that, for the former, the backward pump wave is simply provided by a vertically placed mirror, whereas for the latter the two counter-propagating pump waves are provided by an optical ring-path. It is noted that the first arrangement is much simpler than the second. However, the second arrangement is more convenient to examine the influences from various polarization combinations of three incident beams or to change the intensity ratio among them. To eliminate the possible optical feedback from the backward phase conjugate beam into the laser source, one may put an optical isolator near the output end of the laser device, or simply increase the distance between the laser and the nonlinear medium so that the backward traveling time of the feedback light pulses is longer than the laser-pulse duration.

Various mechanisms that can be used to explain intensity-dependent refractive index changes in nonlinear media, including (a) electronic cloud distortion, (b) intramolecular (Raman) motion, (c) molecular (Kerr) reorientation, (d) optoelectrostriction, (e) photorefractive effect in doped nonlinear materials, (f) population change, and (g) opto-thermal effect [89]. Different mechanisms have different time-response properties and therefore they can be experimentally determined by measuring the rising and decaying behavior of the induced gratings.

7. Photonic Devices:

Much of the work has been done on solutions, clearly unacceptable for any device in the field, unless in thin film form [90]. Both sol-gels and polymers have been proposed and are being actively studied. Unfortunately, their damage thresholds are lower and they could lack a good means for recovery. Liquid just flows to another region and adjust. Currently, most of device development has been on tandem structures or density variations. Hopefully, other device configurations and different solid hosts will be discovered in the near future. The best reported results, that we have seen, have a 500 deduction in signal with a 50% transmission at low intensity with a lead phthalocyanine complex. Clearly, these are some of the most promising materials. Indeed, Perry et. al. [91] reported an attenuation of 540 for a system based on bottleneck idea [92] and using an Indium phthalocyanine. Similar results were reported by Xu et al. [93] with lead phtalocyanine (attenuation of 500) in solid (thin films) and liquid solutions. It is also found that by means of heavy ion irradiation, one can

modify the dielectric and optical properties of some inorganic crystals [94-95] and the same thing can be expected in heavy ion irradiated organic crystals, films, and dye doped polymer films.

8. Materials for Optical Phase Conjugation via FWM:

The most experimental studies on optical phase conjugation (OPC) study using four wave mixing (FWM) technique can be divided into three categories: (1) based on nonresonant media, (2) Four wave mixing based on resonant media, and (3) Four wave mixing based on photorefractive materials.

Degenerate FWM in Nonresonant Media: In this case, a nonresonant nonlinear material (like CS_2) is used for the DFWM experiment. It is found that the spatial resolution of the wave front reconstruction for the phase-conjugate beam generated could be up to 30 lines/mm [96-100].

PCW generation in Resonant Media: Resonant materials which have large third-order nonlinear susceptibility can be used for generation of PCW signals [89]. For this reason, many researchers choose resonant material in which certain types of resonance enhancements of ¡ can be utilized. In early experiments, the metal vapors were used as resonant nonlinear media [101 – 103]. There are many resonant media that can be utilized for degenerate FWM studies with one-photon resonant enhancement [104-110]. It is known that certain dye solutions or dye-doped solid materials have shown very strong linear one-photon absorption or nonlinear two-photon absorption energy bands in appropriate spectral regions, hence, they are good candidates for degenerate or partially degenerate Four Wave Mixing (FWM) studies due to their largely enhanced third-order nonlinearity [110]. Based on literature review, the various types of optical materials commonly used for FWM studies are summarized below [89]:

Absorbing Liquid and Solid Materials: This kind of materials shows a strong one-photon or two-photon absorption [89]. The mechanism causing the induced refractive-index change is either population change or due to opto-thermal effect. The typical materials in this category include dye solutions [111-114], impurity-doped glasses [115-120] dye-doped matrices [121-132], and crystals [133-134], fullerenes (e.g. C^{60}) related materials [135-136], and liquid crystals [137-138].

Lasing Gain Media: Various pumped lasing media such as Nd:YAG and others are used to achieve degenerate FWM and phase-conjugation experiments. One of the mechanisms used to explain the induced gratings in material medium could be the periodic spatial modulation of density distribution. As per published reports, the nonlinear reflectivity currently reached up to ~ 2500 for a small signal intensity and multi-pass geometry [89. 139-144].

Metal Vapors: Four wave mixing can be efficiently generated in various metal vapors, including Na, K, Rb, Cs, and others [143-151]. Compared with liquid or solid absorbing media, metal atoms in the vapor phase shows narrower absorption line width, therefore the frequency of incident laser beams have to be tuned close to the selected spectral transition to reach the expected resonant enhancement. Currently reported nonlinear reflectivity has reached as high as ~ 300 [89, 152].

Photorefractive Materials: Photorefractive materials are extensively studied for their optical phase conjugation properties using degenerate four-wave mixing [89]. The principle of photorefractive effect is the combination of light induced charge separation and subsequent electro-optic response of second harmonic nonlinear material which shows photoconductive property [153-155]. Here, the mechanisms of induced refractive-index change are mostly different from that of third-order nonlinear materials. Hence most of the photorefractive materials used for FWM and phase conjugation studies are impurity-doped inorganic crystals which include $LiNbO_3$, SBN, $LiTaO_3$, $BaTiO_3$, KH_2PO_4, $KNbO_3$,KNSBN, $Bi_{12}(SiGe)O_{20}$,BSO, CdS, GaAs, InP, etc. [156]. In the recent decade, organic crystals and dc-field poled polymer materials have also been used as optimum photorefractive materials for FWM studies [157]. Photorefractive materials can be used to get high efficiency light-induced gratings with low power CW laser beams in OPC experiments [158]. One of the disadvantages of this type of nonlinear material for FWM applications is their slow response time [159].

9. Conclusion:

An extensive literature survey on nonlinear optical materials is depicted in this paper. This includes review on organic nonlinear material research, materials for reverse saturation absorption, molecules for two-photon and multi-photon absorption, experimental techniques for nonlinear refraction, nonlinear absorption, optical limiting and optical phase conjugation. Finally, the various optical device configurations based on nonlinear absorption, nonlinear refraction, optical limiting and optical phase conjugation are discussed along with their advantages and limitations.

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