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Synthesis of transparent wood from lignin modification in *Bombax ceiba*

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Abstract
The uncertainties present in the environment and climate change, and also the rapid non-renewable resource emissions, have driven mankind to create effective energy savers and environmentally friendly sustainable materials. Wood is one of the most abundant and versatile bio-based structural materials. There are several promising and significant benefits of wood, such as its high toughness, low thermal conductivity, low density, high young's modulus, biodegradability, and non-toxic nature. Moreover, while wood has several ecological and structural benefits, it falls short of satisfying the need for optical transparency. High transparency and haze as well as environmental friendliness make transparent wood ideal for use in several important industries, including electronics, packaging, automotive, and construction. Therefore, current research on developing transparent wood is carried out using low-cost materials such as *Bombax* species, balsa and bamboo. Present research highlights the process of making transparent wood along with the change absorbed in the FTIR spectrum during Lignin Modification and polymerization. Later such fabricated transparent wood is subjected to different properties analysis such as density, weight gain, water absorption, flexural strength and transparency.

Keywords: *Bombax ceiba*, low cost, transparent wood, FTIR, optical property, environment friendly

Introduction
The world is fastly running out of fossil fuels (Pang *et al.* 2020) [1], and the need of the hour is focusing much more on renewable sources of energy, Wood has both structural and functional properties, The structural benefits range from the scaffold (Li *et al.* 2018) [2] to building material, and The functional benefits ranges from carbon sequestration (Zeng 2008) to transparent wood. Wood presents numerous structural and ecological advantages but shortfalls in meeting the requirements of optical transparency (Li *et al.* 2016) [5].
Wood is a primary supportive organ essential for producing firm structures since it is a porous and fibrous hard structured stem that also contains the secondary xylem of the vascular tissue made of cellulose, hemicellulose, and lignin (Jin *et al*. 2017) [6]. Wood has had multifunctional potential since ancient times due to its structural and functional features, and it is still commonly employed in building (Kuzman & Groselj 2012; Wimmers, 2017) [7, 8], fencing (Guzel, 2020) [9], utensils (May 1950) [10], and hand tools preparation (Shirai, 2021) [11]. Lignin is a complex and recalcitrant phenolic macromolecule comprising phenylpropane type units that provides cell walls stiffness even in absence of turgor pressure.
Its highly irregular polymeric structure and very low solubility in common solvents make, it difficult to lignin isolation from wood and its conversion to value-added products (Miedes, *et al.* 2014) [12]. Lignin is usually seen in the secondary cell wall, but the middle lamella and cell wall corners have a high concentration (Planta, 2006). Mainly wood chromophores are located on lignin (Heitner, 1993) [13], These chromophores in lignin have the carbon-carbon double bonds conjugated with the aromatic rings, along with other aromatic extractives, contribute to the dark color of wood and lignin is responsible for around 80–95% of light absorption in wood (Wu *et al.* 2020) [14].
In Transparent wood, the principle lies in Removing this lignin and impregnating the lignin removed template with a matching refractive index polymer to increase the transparency. Thus prepared wood has many ample advantages, ranging from smart windows to smartphone screens, and rooftops to interior aesthetic panels. Transparent wood building material can contribute to less indoor fossil fuel energy consumption by replacing it with thermally

insulating material for promoting effective sunlight harvesting in place of artificial lights (Samanta *et al*. 2021; Mi *et al*. 2020a; li *et al*. 2019) [15, 17,]. Gan *et al*. (2017) [16] prepared Magnetic transparent wood by incorporating ferromagnetic (Fe_3O_4) nanoparticles. By adding Si and CdSe/ZnS core/shell quantum, Demir *et al*. (2011) [19] fabricated Luminescent transparent wood with enhanced diffused luminescence, and the luminescence diffusivity was controlled by the light scattering of the transparent wood. Fu *et al*. (2017) [18] used nano clay impregnation into the cell wall for fire retardancy and Bisht *et al*. (2021) [20] developed a UV resistant transparent wood for outdoor application by incorporating epoxy resin doped with a UV absorber (2-(2H-Benzotriazol-2-yl)-4, 6-di-tera-pentylphenol) (conc. 1.0 and 1.75% w/v) respectively)

According to the European Union, even small environmental conservation steps like 1% fossil fuel reduction may save 60 billion dollars per year (Scott & Gössling, 2021) [21] in energy consumption, thus gradual replacement of fossil fuels with bio-based materials plays an important role. Henceforth transparent wood is the major boon and breakthrough step in innovation towards a circular economy (Wu *et al.* 2020) [14].

Bombax ceiba, one of them, belongs to the family Bombacaceae which contains about 30 tropical genera and 250 species. The plant species is used in different systems of medicine in India, China and Southeast Asian countries too. Every part of the plant served not only as medicine but also used for various commercial purposes such as fodder, fuel fiber and ship/ boat/catamaran building now demonstrated for making as a transparent wood composite (Maurya *et al* 2018) [3].

Materials and Methods

Bombax (*Bombax ceiba)*

During the work, the *Bombax* (*Bombax ceiba)* is sourced from the forest college and research institute wood processing unit.

Sodium Hydroxide (NaOH)

In the work NaOH will be used as one of Lignin Modification chemical. It is a bleaching agent which will be effective in removing the partial removal and modification of lignin. The NaOH will be supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana.

Hydrogen Peroxide (H_2O_2)

In the study H_2O_2 will be used as one of Lignin Modification chemical. It is an effective bleaching agent which is used in partial removal of lignin and modification of chromatophores in Lignin. The Hydrogen Peroxide will be supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana.

Acetone (CH_3COCH_3)

In the preparation CH_3COCH_3 will be used as one Moisture Cleansing agent. It is effective in removing the moisture and other chemical present in the wood. The Acetone will be supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana.

Ethanol (CH_3CH_2OH)

In the study Ethanol will be used as one of Moisture Cleansing agent. It is effective in removing chemical present in the Lignin Modified wood as it is good volatile liquid. The Ethanol was supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana.

Disodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2Na_2O_8$)

In the study Disodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2Na_2O_8$) was used as one of Lignin Modification chemical. It is an effective chemical which was used in modification of Lignin in wood. The Disodium ethylenediaminetetraacetate dihydrate will be supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana.

Magnesium Sulphate ($MgSO_4$)

In the work $MgSO_4$ will be used as one of Lignin Modification chemical. It is an effective chemical which will generally used in modification of Lignin in wood. The magnesium sulphate will be supplied by Tarnaka chemicals private Limited, 1-91, Habsiguda x roads, Hyderabad 500 007, Telangana

Hot Plate Magnetic stirrer

During the Preparation Hot plate Magnetic stirrer instrument will be used for boiling the sample in Lignin Modification. Lignin Modification is an important step in Modifing the lignin present in wood samples. The Neuation iStir HP 350 instrument is supplied by neuation technologies pvt ltd, Plot No. 15 GIDC Electronics Park SEZ Kolavada Road, Sector - 26 Gandhinagar, Gujarat - India PIN: 382026.

Vaccum Pump

During the Polymerisation process Vaccum Pump will be used for effective infiltration of polymer into the wood template. Polymerisation is an important step in preparing the transparent wood. The ARSH 2 stage Vacuum pump instrument is supplied by B No. - 4,1,37, Sahara Complex, Hanuman Tekdi, Abids, Hyderabad, Telangana 500001.

Electric Balance

During the work electric balance will be used for measuring weight of samples for different samples. The electric balance is supplied by Shimadzu Analytical (India) Pvt. Ltd. A/B, Rushabh Chambers, Makwana Road, Marol, Andheri (E), Mumbai - 400 059.

Epoxy

During the preparation of Transparent wood, an industrial grade of Epoxy Primer and Hardener of 3:1 is used as the polymer, The grade of epoxy plays an important role in optical properties of transparent wood. The Epoxy will be supplied by virtuoso apps private limited, Erragadda, Hyderabad - 500018.

Method

The transparent wood was generally prepared by two continuous processes such as Lignin removal or Lignin modification and the Polymerization Process.

Lignin Modification (LM)

In this research, one of the main objectives that were given much emphasis was the modification of lignin by chemical treatment. This step was taken to ensure the that maximum light absorption component in wood get modified or partially removed. The experimental works of the modification process are explained following steps.

Pre-treatment of Lignin Modification

Before the Lignin Modification the samples are sourced from different location as mentioned in Material section. Then the samples are processed into various dimension as required for various treatments in Wood Processing workshop of forest college and research institute.

Preparation of Lignin Modification (LM) solution

Different solution required for the Lignin modification are prepared before carring out lignin modification process, In that first 10 Wt% NaOH solution was prepared by mixing 10 g of NaOH pellets in 100ml of Distilled water, Then after 1gram of Magnesium Sulphate ($MgSO_4$) and Disodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2Na_2O_8$) was mixed in 100ml of distilled water to make 0.1 Wt% of Solution., Later with 4 Wt % of H_2O_2 solution was prepared by diluting Concentrated 30Wt% H_2O_2 by adding Distilled water before the Lignin Modification process.

Lignin Modification by using sodium Hydroxide and Hydrogen Peroxide Chemicals

During the stage of modification, the NaOH salt was added into distilled water which was the solvent in this process at a ratio of 10 g of NaOH to 100 ml of Distilled water and the samples of *Bombax* veneer of 4.5 cm X 2 cm in length and width having 1mm thickness respectively were immersed in NaOH solution and placed on Hot Plate for 10 minutes, Later 4 Wt% H_2O_2 was added at 80 °C and subsequently 0.1 Wt% $MgSO_4$ and 0.1 Wt% $C_{10}H_{14}N_2Na_2O_8$ were added into beaker, Thereafter samples were heated until they turns white as shown in Fig.1

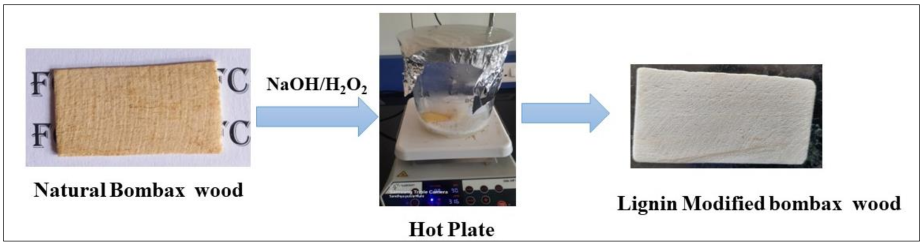

Fig 1: Lignin Modification *Bombax* wood sample from Natural wood *Bombax* sample

Then Lignin Modified wood pieces were taken out and placed in hot distilled water in order to exchange the chemical residues added in it, during lignin modification process. Then these wood pieces were immersed in acetone and Ethanol solution to remove the moisture in the wood for effective impregnation of polymer into it.

Fabrication of Epoxy-based Transparent Wood

After a successful Lignin modification process, The Transparent wood is prepared by using lignin modified template, Epoxy, and Vacuum Pump. The stages involved in the Transparent wood fabrication process are as described below:

Mixing

During this process the epoxy resin and Hardener was taken into Petri dish in the ratio 3:1 mix ratio by volume, (3-parts resin to 1 part hardener) and mixed it manually. Then resin is mixed until the air bubbles disappear in the resin as shown in Fig.3.2 Removing of Air bubbles was important as they enter into the transparent wood during polymerisation stage.

Polymerisation

During the Polymerisation process, the lignin-modified dehydrated wood samples are taken out and placed in vacuum for few minutes to remove acetone in it. Later this sample were immersed in the Epoxy resin of having similar refractive index.

The amount of pressure applied with time of vacuum infiltration, and stabilization of LM wood plays important role in the impregnation of epoxy into the wood. At first, to stabilize the wood in the vacuum chamber, the wood was immersed in the epoxy solution. During the stabilization, i.e. when the wood was placed in epoxy solution is under vacuum, the air that is present in the pores of the wood is released as bubbles Once, the wood Epoxy solution is stabilized and filled in Wood Lumen, the chamber was turned off and retained it under vacuum.

Then epoxy impregnated wood was then further sandwiched in between the glass slides with wrapping aluminum foil and placed in hot air oven at 85 °C until the resin hardens. This cured wood is then taken out and labeled as transparent *Bombax* wood as shown in fig 2.

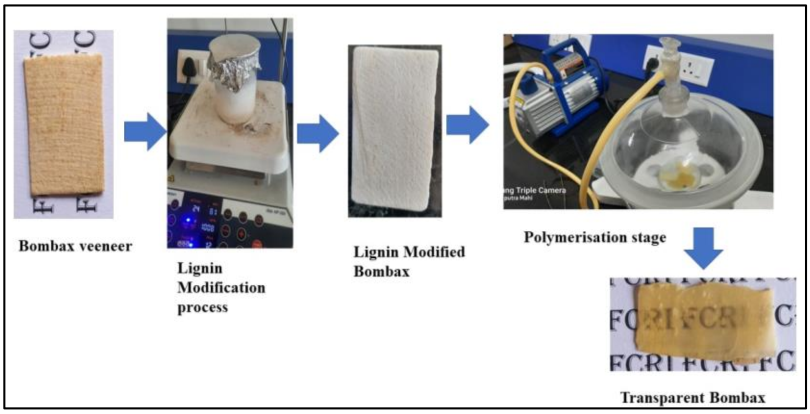

Fig 2: Fabrication process of Transparent wood

Physical properties
During the study the physical properties analyses that were carried out on the TWC were density, weight gain percentage and water absorption rate.

Density
Measurement of density was conducted according to ASTM D 792. Specimens were dried for 24 hrs at 105 °C. The value of the density was calculated using the following equation:

$$\rho = \frac{md}{V} \quad (3)$$

where ρ is the density, v is the volume and m_d is the dry of specimen.

Weight gain test
Weight gain test was carried on samples to study the weight gain behavior of sample after polymerisation. The calculation of weight was done by electric balance Model-AP225W-AD, from forest college wood science and technology lab which was provided by Shimadzu. Specimens were dried for 24 hrs at 105 °C. The value of the density was calculated using the following equation:

$$W=M_a - M_b /M_a \times 100\% \quad (3.1)$$

In formula, W represents weight gain rate, M_a represents mass after impregnation, M_b represents mass before impregnation.

Water absorption
Water absorption analysis was carried out on sample of composites so as to study the moisture absorption capacity of composites reinforced with polymer. In addition, to examine the effect of different types of wood species on water absorption capacity of transparent wood composites. The water absorption (WA) test was conducted in accordance with ASTM D 570. The weight of each specimen was measured before testing and conditioned samples of each composite type were soaked in distilled water at room temperature for 1-3 days. Samples were removed from the water, patted dry and then measured again.
The value of the water absorption in percentage was calculated using the following equation:

$$MB = W_f - W_i / W_i \times 100\% \quad (3.2)$$

Where MB is the water absorption rate
Where W_f -Weight of sample after absorption,
W_i- Weight of sample before absorption

Mechanical properties testing
In this work, the mechanical property test that were done on the Transparent composites samples was flexural testing. Flexural strength and flexural modulus were obtained from this flexural testing.

Flexural testing
Flexural testing otherwise referred to as 3-point bending test was carried out to determine the force required to bend the composites sample under a 3-point loading, as well as its resistance to elastic deformation under an applied force (flexural modulus). Flexural testing was carried out in line with ASTM D790-97 standard using an Mecmesin Multi-test 10i universal testing machine (UTM) from IISc Bangalore with static load cell of 5 KN. The dimensions of test sample were 65 mm x 3.4 mm x 10 mm and cross head speed was set at 2 mm/ min with support kept at 20 mm apart. testing was done at room temperature.
Flexural strength can be calculated using the relation below:

$$\sigma = \frac{3\,FL}{2\,bd^2} \quad (3.3)$$

Where, *F* = Load (Force) at fracture point, *L* = length of support span, *b* = width of test sample and *d* = thickness of test sample.

Fourier transforms infrared spectrophotometer (FTIR)
In this study, FTIR analysis was used to examine the structural and functional groups present into the transparent wood samples. This functional group analysis was carried out with the help of Perkinelmer Frontier Model Fourier transforms infrared spectrophotometer using the standard ATR technique from Indian institute of science IIsc Bangalore. The samples were scanned on FTIR spectrometer. The average of more than 10 scans were taken at 2cm^{-1} resolution from 600 to 4000 cm^{-1}.

Optical property
In this study, ultraviolet-visible spectrophotometer analysis was carried out on TWC and Natural Wood species to observe the optical properties of samples. Optical properties are measured according to ASTM D1003 ("Standard method for haze and light transmittance of transparent plastics"). The samples were run at 400-700nm using an ultraviolet-visible spectrophotometer (Shimadzu UV 3600) instrument from the Indian Institute of Science, Bangalore.
The transmittance of the sample was calculated by using formulae as follows.

$$\text{Transmittance} = T2/ T1 \times 100\% \quad (1)$$

Where T1 is the incident light,
T2 is the transmitted light of the sample.

Results and Discussions
Structural Characterizations
In this study, Fourier Transform Infrared (FTIR) spectroscopy was employed to identify whether the Lignin Modification chemicals removed or modified lignin present in the wood. The chemical changes that occurred upon the incorporation of Lignin modification chemicals into the wood are presented in Figure 4.3. The Infrared absorptions bands that are commonly seen in wood were observed in all six samples with few changes. FTIR by attenuated total reflection (ATR) method was generally used to determine the functional groups of transparent wood. The wood composition of cellulose, hemicellulose, and lignin have several functional groups such as hydroxyl (O–H), carboxyl (C–H), carbonyl, methyl groups (CH_3), benzoyl groups etc, which are generally analysed by FTIR spectrum. The characteristic of broad peak that were observed at 3200-3460 cm^{-1} are attributed to the hydrogen bonded (O-H) and O-H stretched in the spectrum of transparent wood composites TWC
Wu *et al.* (2019) [22] noted that FT-IR spectra of natural wood samples generally indicate the peak of 3334 cm-1 for O-H bonding, 2897 cm-1 for C-H stretching vibration, 1734 cm -1

for C=O stretching vibration, and 1593,1502 cm-1 for C=C stretching vibration. These bands were related to the hydroxyl groups in celluloses, carbonyl group of acetyl ester in hemicelluloses, and carbonyl aldehyde groups in lignin. The tentative assignments of the main absorption bands of the FTIR analysis during Lignin Modification are illustrated in Table 4 as noted by Wu *et al* 2019 [22].

Table 1: Tentative FTIR peaks Assignments of Transparent wood composite

Wavenumber (cm^{-1})	Assignment
3334	Hydrogen bonded (O-H)
2897	Stretching of C-H
1734	Free C= O
1593	C=C
1234	C–O

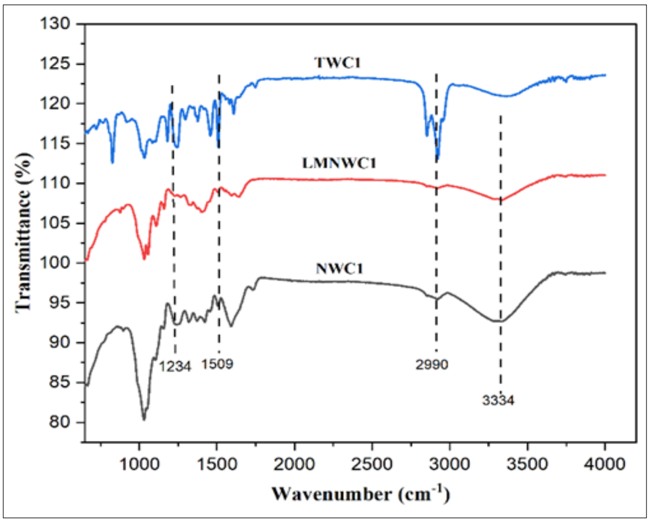

Fig 3: FTIR spectra of TWC1, LMNWC1, and NWC1 (NWC1-Natural Wood *Bombax*, LMNWC1-Lignin Modified Natural wood *Bombax*, and TWC1-Transparent Wood *Bombax*)

From Fig.3 Partial removal of lignin and elastic vibration of the lignin aromatic ring skeleton was indicated at during 1509 cm^{-1} in LMNWC1. Reduction in bands for LMNWC1 at 1234 cm^{-1}spectra is attributed to unconjugated C=O and C–O indicating the partial dissolution of hemicelluloses during the treatment as supported by Rao *et al*. (2019). The weakening of peak 3334 cm^{-1} suggests that lignin was partly eliminated as noted by Lu *et al*. (2020). The peak at 2990 cm^{-1} indicates C-H stretching vibration as mentioned by Wu *et al*. (2019) [22]. From above fig 3 data we can conclude that lignin Modification happened in LMNWC1.

Density

Bombax showed density of 347.14 kg/m^3 with its weight of 0.62g, upon imprgnation of polymer into *Bombax* it improved its density to 677.21 kg/m^3 by adding weight to 1.21g

Weight gain

The *Bombax* spexies showed weight gain from 0.62 g to 1.21g which accounts 95.04% of its intial weight

Water absorption rate

The natural *Bombax* species showed a water absorption rate of 206.99% and whereas transparent *Bombax* showed rate of 21.01% for a duration of 72hr water soaking. The less absorption rate in transparent wood is attributed to good hydrophobic behaviour nature of epoxy as noted by Bisht *et al.*, (2021) [20]. It is also concluded that due to successful curing epoxy in matrix also left few cells for water to imbibe as noted by Mi *et al.*, (2020) [17].

Flexural strength

Initial flexural strength of *Bombax* species is 36Mpa, it is improved to 77Mpa upon modifying the wood into transparent. This is generally attributed to the filling of smooth plant tissue with hard resin material, but the intensity of strength depends on the internal compatability of matrix and hydrophobic resin material.

Optical property
Transmittance

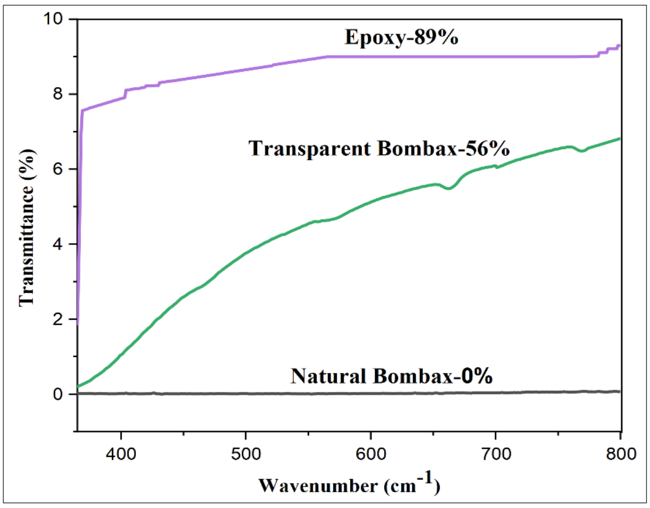

Fig 4: Transmitance of Norma *Bombax*, transparent *Bombax* and Epoxy resin

From Fig 4. The Epoxy polymer showed a transmittance of 89% in the range of visible spectrum 400 to 700 nm, the Natural *Bombax* sample showed a transmittance of 0% throughout the range whereas the *Bombax* samples with modified lignin structure impregnated with epoxy resin showed transmittance of 56% and reaching the reference epoxy resin transmittance as seen in Fig 4.

Conclusion

Density improvement is because of resin impregnation which added weight into the species with less change in its volume. The less percentage change in water absorption rate in TWC can be concluded that epoxy is better impregnated. *Bombax* showed strong interfacial interactions in the composite, which resulted in high flexural strength of 77 MPa by transferring effective stress from matrix to fibers

Fourier transform infrared spectroscopy (FTIR) showed that the *Bombax* had lignin modification and partial elimination of lignin at 1509 cm^{-1} at 33340 cm^{-1} respectively, and impregnating this template with epoxy showed optical transparency of 56%.

It can be concluded that the use of H_2O_2 and NaOH as lignin modification chemicals, modified less lignin in *Bombax* as it showed transmittance of 56%. The above low transparency was also attributed to low mismatching of refractive index of matrix and polymer.

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