Synthesis and characterisation of curcumin–M (M = B, Fe and Cu) films grown on p-Si substrate for dielectric applications

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Metal-coordinated yellow curcumin was extracted from green natural sources and sublimated in vacuum to prepare thin films on p-Si and glass substrates for dielectric and optical investigations. The synthesized curcumin complexed with the metals boron, iron, and copper powders were crystalline while the prepared films were amorphous. The optical absorption spectrum of the prepared films showed similar two absorption band structure in the visible range. The onset energy of the main optical absorption band of the film was determined using the Tauc technique. The dielectric properties of this material were systematically studied for future applications in metal-insulator-semiconductor MIS field of applications. The complex dielectric properties were studied in the frequency range of 1–1000 kHz and was analysed. The important find is a large optoelectronic sensitivity so that the integral optical responsivity (S) reaches ~1.0 A/W and the electrical conductivity increases under light illumination by ~400–1000%. Generally, Curcumin metal complex can be used in small-k environmentally friendly production of microelectronic and optoelectronic devices.

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1. Introduction

Curcumin [1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione] of chemical formula C21H18O6 is a yellow pigment extracted from the rhizome of the plant Curcuma longa L. The powdered rhizome of this plant is a spice called turmeric. Some general physical properties of curcumin are well-known for example it is insoluble in water but soluble in organic solvents like methanol and acetone. Recently, the chemical and medical properties of curcumin was surveyed in Ref. [1]. Traditionally curcumin is used as a flavouring agent in curries and as a food additive. Also, it has been investigated in medicine as an anticancer, antiviral and anti-inflammatory agent [3–8]. This anticancer properties of curcumin have generated the greatest interest, resulting in many publications. However, curcumin is also interesting from scientific, industrial, and economical point of views; to use such material from direct plant source in the electronic, solar, and optoelectronic applications. The optical study of curcuminoid shows that it has an absorption peak at ~430 nm in linear absorption spectrum [1] and has a large value of negative nonlinear refractive index.

Generally, the physical properties of curcumin have not been well investigated. On other side, it is possible to insert metallic ions inside the molecular structure of curcumin forming “curcumin–M” complex as an organometallic complex. The metallic ions embedded in organometallic complexes play a role of attraction centre in the molecules through strong metal–ligand covalent bonds [2]. In general, the organometallic complexes have recently attracted considerable interest in electronic, solar, and optoelectronic applications. Our present work focuses on synthesis of curcumin–M complexes and investigates their physical properties such as insulating, electrical and optical properties; these properties yet have not been studied. The present work treats these complexes as insulating and optical materials that might be used in future “green” applications.

2. Experimental details

2.1. Synthesis of curcumin–M

The curcumin–M complex has been synthesized from reagent grade chemicals. Curcumin [1,7-bis(4-hydroxy-3-methoxy-phenyl)-1,6-heptadiene-3,5-dione] was synthesized from vanillin and acetyl acetone using boric acid as a protecting group and 1-butylamine as a base in a solvent mixture of toluene and dimethylformamide. All materials used for synthesis of curcumin–M are of...
puriss grade as purchased and no further purifications were done. The synthesized curcumin was subsequently complexed with boron, copper and iron respectively. The general synthetic procedure of the metal complexes (curcumin–M) involved dissolution of synthesized curcumin in methanol followed by addition of an aqueous solution of the relevant metal chloride or acetate. In the synthesis of the boron and copper complexes the reactant molar ratios were curcumin: metal 1:2. With regard to the iron complex iron a ratio of curcumin: iron 1:3 was used. The mixture was refluxed for a period of 3 h followed by cooling. The precipitated complexes were filtered washed with cold water, followed by ethanol, then dried and purified. The molecular structures of the metal complexes of curcumin are shown in Fig. 1.

2.2. Measurements

A set of thin films of each of the curcumin metal complexes of B, Fe, and Cu were slowly deposited (about 0.1 nm/s) by thermal sublimation in a vacuum system of about 10⁻³ Pa on chemically (using HF) cleaned p-Si(100) wafers and ultrasonically cleaned corning 2947 glass substrates held at room temperature. After deposition, the thickness of the prepared complex films were measured by an MP100-M spectrometer (Mission Peak Optics Inc., USA). The films were characterised by X-ray diffraction (XRD), optical absorption spectroscopy, and electrical measurements. The crystalline structure was investigated with a Rigaku Ultima VI 0–2θ X-ray diffractometer equipped with Cu Kα radiation (0.15406 nm) and operating at 40 kV and 40 mA. The spectral normal transmittance T(λ) and reflectance R(λ) in the transparent and absorption regions (300–1500 nm) was measured by a UV–Vis–NIR Shimadzu UV-3600 double beam spectrophotometer. The electrical measurements on the prepared films as insulators were carried out on samples prepared in form of metal–insulator–semiconductor MIS structures by using aluminium thin film electrodes of about 150 nm and gate area of 4π mm². The ac-electrical measurements were performed using Keithley 3330 LCZ and hp 4275A LCR instruments in a frequency range of 1–1000 kHz with a signal of 50 mV and the dc-measurements were carried out with a Keithley 614 electrometer. The capacitances have been corrected for the stray capacitance.

3. Structural and optical characterisation

Fig. 2 shows the XRD patterns of the synthesised curcuminoid–M (M = B, Fe, Cu) powders together with pure Curcumin for comparison. The patterns were indexed according to the monoclinic (P2₁/n) crystalline structure of lattice parameter (Table 1) close to the known parameters: a = 1.2707 nm, b = 0.72186 nm, c = 1.9880 nm, α = 90°, β = 95.348°, γ = 90°, volume Vcell = 1.81559 nm³ [9]. The parameters were refined with by using Rietveld method with PDXL program. The parameters are slightly increased with by inserting ion so that Vcell increases with increasing of embedded ionic radius [10], as shown in Fig. 3. Fig. 2 reveals almost similar crystallinity for the synthesised powders except that of Cur–Cu which suffered crystal deterioration, due to high Cu²⁺ ionic radius that increases the molecular size. However, all the prepared thin films were amorphous, as shown in the inset of Fig. 2, although we tried unsuccessfully to crystallize them by
The spectral (double beam) normal transmittance \( T(\lambda) \) and reflectance \( R(\lambda) \) are used to evaluate the spectral absorption coefficient \( \alpha(\lambda) \) by \( \alpha(\lambda) = (1/[\ln(1 - R(\lambda))]T(\lambda), \) where \( d \) is the film thickness [14]. The optical absorption onset \( (E_{\text{onset}}) \) can be evaluated by using the Tauc technique [15]:

\[
\alpha(\lambda) = \frac{C_{\text{op}}(\hbar \nu - E_{\text{onset}})^n}{\hbar \nu}
\]

where \( \hbar \nu \) is the photonic energy and \( C_{\text{op}} \) is the film’s constant. The exponent \( n \) is equal to 0.5 or 2 for direct or indirect optical transitions, respectively. It was found a linear graphical relationship for \( (\alpha(\lambda)\lambda)^2 \) vs. \( \lambda \) as shown in Fig. 5, by which the \( E_{\text{onset}} \) could be determined to be 2.49 eV, 2.50 eV, and 2.55 eV for Cur–B, Fe, and Cu, respectively. The value of \( E_{\text{onset}} \) slightly blue shifted with increasing of ionic mass number of embedded ion (the estimated error in the present method is \( \pm 0.02 \) eV).

The refractive index of the Cur–M films can be calculated by using the following formula for transparent films:

\[
n = \left( 1 + R_f \right) / \left( 1 - R_f \right)
\]

where \( R_f \) is the reflectance of the film, which can be found from the measured experimental reflectance \( (R_m) \) of film/glass substrate (Fig. 4) with the following approximated relation [16]:

\[
R_f = R_m - \left( R_m/T_m^2 \right)T_m
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R_f = R_m - \left( R_m/T_m^2 \right)T_m
\]
where $R_i$ is the reflectance of the glass substrate that was measured experimentally from the used naked glass substrate, $T_i$ is the used glass substrate transmittance, and $T_m$ is the measured experimental transmittance of the film/glass substrate. The spectral calculated refractive index $n(\lambda)$ is given in Fig. 6. The refractive index at the long wavelength of 1500 nm is 1.38, 1.47, and 1.40 for Cur–B, –Fe, and –Cu, respectively, i.e. the average is 1.41; in agreement with known value for pure curcumin [17,18]. The calculated spectral refractive index $n(\lambda)$ has a maximum value at about 492 nm, 485 nm, and 450 nm, for Cur–B, –Fe, and –Cu, respectively; i.e. blue-shifted by increasing the mass of the coordinated ion. We have to mention that this value was about 505 nm for pure curcumin given in Ref. [12]. The found spectral refractive index can be fitted to the well-known single oscillator Wemple–DiDomenico (W–M) model [19]:

\[
1/(n^2 - 1) = (E_0/E_d) - (1/E_0E_d)/(h\nu)^2
\]

where $E_0$ is the energy of the effective dispersion oscillator, $E_d$ is the dispersion energy. The linear relationship between $(n^2 - 1)^{-1}$ and $(h\nu)^2$ in the transparent region is shown in the inset of Fig. 6. The calculated pair of parameters $(E_0, E_d)$ are (2.15, 1.68), (2.92, 3.13), and (4.8, 3.88) eV for Cur–B, –Fe, and –Cu, respectively; i.e. these parameters increase by increasing the mass of the embedded ion.

### 4. Dielectric properties

#### 4.1. AC properties

The ac-measurements were done on samples constructed in the form of MIS structure (Al/Cur–M/p-Si), which was characterised by the well-known method of measuring the high-frequency (1 MHz) gate voltage dependence of capacitance ($C(V_g)$). The flat-hand voltage for all samples ($V_{th}$) was $\sim -1.3$ V and the fixed charge density ($Q_{rms}$) in the structure was estimated to be $(3-4) \times 10^{10}$ charges/cm$^2$ by using $Q_{rms} = (C_{mss}/q)(\Phi_m - V_{th})$, where $C_m$ is the accumulation capacitance per unit area and $\Phi_m = -0.9$ V is the work-function difference between Al gate and the used p-silicon substrate of an acceptor concentration $\sim 1 \times 10^{16}$ cm$^{-3}$ [20,21]. These fixed charge-centres are created due to various types of defects and impurities in the insulator and on I/S interface. The values of $Q_{rms}$ are in the usual device grade ($10^{10}-10^{11}$ charges/cm$^2$) [22], and it is not high enough to pin the Fermi level of the Si substrate [23]. Furthermore the gate–voltage ($V_g$) dependent of $C^{-2}$ [21] gives the barrier height of a Schottky diode to be $\Phi_m = -1.3$ V for all the studied samples, as shown in Fig. 7.

The dielectric properties of the Cur–M as insulators were studied in the frequency range 1–1000 kHz at accumulation gate voltage of $-1.3$ V. The frequency dependence of the dielectric constant $\varepsilon'(\omega)$ and dielectric loss $\varepsilon''(\omega)$ are shown in Fig. 8. It is clear that $\varepsilon'$ decreases with increasing frequency getting at 1 MHz the values $\sim 2.0, 1.7$, and $2.3$ for Cur–B, –Fe, and –Cu, respectively. However, the type of $\varepsilon'(\omega)$ dependent for Cur–B and Cur–Cu insulators are identical but different from that of Cur–Fe insulator reflecting different micro structure of the films that might be due to the magnetic nature of embedded Fe ions, which cause some molecular agglomeration with boundaries. Materials with such small-$k$ values can be used in the environmentally friendly production of microelectronic devices.

The dielectric loss $\varepsilon''(\omega)$ function has a minimum in the studied frequency range, as shown in Fig. 8. Physically, such behaviour is caused by the effect of a distribution of relaxation times around some mean value $\tau_m$. Moreover, such behaviour can be modelled by assuming that the equivalent circuit of the investigated capacitor consists of three elements: a pure eigen–capacity element ($C_e$),
connected in parallel with an inherent resistance element ($R_s$) and a small series resistance ($r$) of leads and electrodes; consequently this gives, $\omega_{\text{min}} = \left( r R_s e^2 \right)^{-0.5}$ [24]. The value of $f_{\text{min}}$ is 29.8 kHz, 5.8 kHz, and 24.5 kHz for Cur–B, –Fe, and –Cu, respectively; the value of $f_{\text{min}}$ for Cur–Fe is much lower than that of Cur–B or Cur–Cu that could be due to the magnetic properties of Fe embedded ion, which can control the microstructure of the grown Cur–Fe film.

The ac-conductivity ($\sigma_{ac}$) of insulators is often expressed by the following relation: $\sigma_{ac} = \sigma_{ac}(0) + \sigma_{ac}(\omega)$, where $\sigma_{ac}(0)$ is the dc-conductivity of the insulator and $\sigma_{ac}(\omega)$ is the frequency-dependent component. $\sigma_{ac}(\omega)$ is typically expressed by a power law $\sigma_{ac}(\omega) = A_\omega \omega^s$, where $A_\omega$ is a coefficient [25]. According to the correlated barrier hopping (CBH) model, the exponent $s$ is correlated to the effective barrier height for hopping $W_M$ that has any value smaller than the bandgap ($E_g$) and hence $s < 1$ [26]. $W_M$ should depend on the film's microstructure as the average grain size, texture orientation, defect distribution, phase content, etc. However, none of these factors was entered into the (CBH) model derived originally for uniform homogeneous non-crystalline insulators. Therefore, the experimental value of $s$ could take any value <1 or >1, as it was observed experimentally [27,28]. The results of $s > 1$ were explained by the non-random distribution of hopping centres because of the inhomogeneity in the structural defect distribution that enhances the exponent “$s$” by an additional factor [26]. Fig. 9 shows the frequency dependence of ac-conductivity $\sigma_{ac}(\omega)$. The application of the above model gives values of the pair ($\sigma_{ac}(0), s$) as: $(1.23 \times 10^{-9} \text{S/cm}, 1.33)$, $(3.75 \times 10^{-9} \text{S/cm}, 0.98)$, and $(2.38 \times 10^{-9} \text{S/cm}, 1.26)$ for Cur–B, –Fe, and –Cu, respectively. The small value of $\sigma_{dc}$ ($10^{-9} \text{S/cm}$) refers to that the curcumin are good insulators.

4.2. DC-leakage properties

The $I$–$V$ characteristics of Al/Cur–M/p-Si, MIS diodes were measured and the results are shown in Fig. 10. The diodes exhibit rectifying characteristics with rectification ratio (RR) at ±5 V of $\sim$1.11 $\times$ 10$^3$, 1.0 $\times$ 10$^3$, and 2.2 $\times$ 10$^3$ for Cur–B, –Fe and –Cu, respectively. The diodes, in general, exhibit two conduction mechanisms operating in various forward bias voltage ranges. The low forward gate voltages mechanism is taken place at $V_g < 0.5$ V, where the forward current of the diode increases exponentially with increasing biasing voltage. Therefore, it can be evaluated that Al/Cur–M/p-Si, MIS behaves like a Schottky diode. Thus, the forward $I$–$V$ characteristics of the diode for $V > k_B T/q \approx 0.075$ V can be analyzed on the basis of thermionic theory by the following relation [21,29]:

$$I = I_0 \exp\left(\frac{qV}{n_0 k_B T}\right) - 1 \quad (5)$$

where $n_0$ is the diode ideality factor, $q$ is the electronic charge, $k_B$ is the Boltzmann constant and $I_0$ is the reverse saturation current, $I_0 = A A^f \exp(-q\phi_{BO}/k_B T)$, where $A$ is the area of rectifying contact, $A^f$ is the Richardson constant ($A^f = 32 A/cm^2 K^2$ for p-Si [20]), and $\phi_{BO}$ is the effective barrier height at zero bias. The value of $n_0$ was determined from the slope of the straight line in the semilog plot: $\ln(I)$ vs. $V_g$ for $V_g < 0.5$ V i.e. $n_0 = (q/k_B T) (dV_g/d\ln I)$ to be 3.0, 2.5, and 2.9, for Cur–B, –Fe, and –Cu, respectively. The obtained $n_0$ value is higher than unity due to the existence of the insulating Cur–M layer as interfacial layer between metal (Al) and semiconductor (Si). The value of $\phi_{BO}$ was calculated from $\phi_{BO} = (k_B T/q) \ln(A A^f /I_0)$ with the average $I_0 = 1.2 \times 10^{-3}$ A, to be $\sim$0.82 eV in the average for all samples. A series resistance at zero bias voltage is one more basic parameter describing the diode and can be calculated from the Norde equation [30] $R_s = |k_B T/q| (\gamma - n_0) |I_0|$, where $\gamma$ is the first
integer (dimensionless) greater than \( n_e \). Thus, \( R_i \) is in the range of \( 10^9 - 10^{12} \) \( \Omega \).

In order to determine the conduction mechanism for the leakage current in the studied insulators, the voltage across the insulator layer \( V_{ins} \) was approximated by \( V_{ins} = V_f + \varphi_{ms} \), where \( V_f \) is the value of forward voltage. Thus, we study the leakage current at higher forward bias voltages i.e. for voltages in the accumulation region of magnitude \( V_f > (1.3-0.9) \) \( = 0.4 \) \( V \), where the insulator properties control the conduction rather than the depletion region.

Several mechanisms have been proposed to describe the dc-leakage current transfer through insulators. Among them are the space-charge-limited conductivity (SCLC) mechanism, Richardson–Schottky emission (RS), and Poole–Frenkel mechanism (PF). The relationship between the current density \( J \) and the applied voltage across the insulator layer \( (V_{ins}) \) at temperature \( T \) is given by Eq. (1) for RS mechanism: \( [31,32] \):

\[
J = A_T^2 T^2 \exp\left\{\left(\frac{\beta_{in} V_{ins}^{0.5}}{d^{0.5} - \Phi_{in}}\right) / k_B T\right\}
\]

where \( A_T \) is the effective Richardson constant, \( \Phi_{in} \) is the RS barrier height, \( k_B \) is Boltzmann constant, and \( d \) is the film thickness, which was 124 nm, 135 nm, and 192 nm for Cur–Fe, Cur–B, and Cur–Cu, respectively. The field-lowering coefficients, \( \beta_{in} \) is given by: \( \beta_{in} = (e^2/4\pi \epsilon_0 e_{dyn} d^{0.5}) \), where \( \epsilon_{dyn} \) is the dynamic permittivity of the insulating layer. Fig. 11 shows the measured room-temperature \( I(V_{ins}) \) dependence in accordance with RS model: \( \ln(I) \) vs. \( V_{ins}^{0.5} \) for MIS structure at accumulation polarity. In general, two voltage regions can be observed following different mechanisms. Data for \( V_{ins} < 1.5 \) \( V \) follow RS model showing as solid lines. Data analyses give values of the pair \( (\beta_{in} \) and \( \epsilon_{dyn} \) \) as: \( (2.63 \times 10^{-5} \) \( \text{eV} m^{1/2} V^{-1/2} \), \( 2.07) \), \( (2.86 \times 10^{-5} \) \( \text{eV} m^{1/2} V^{-1/2} \), \( 1.76) \), and \( (2.50 \times 10^{-5} \) \( \text{eV} m^{1/2} V^{-1/2} \), \( 2.2) \) for Cur–B, –Fe, and –Cu, respectively. To within the estimated experimental error \( (\Delta \beta/\beta \approx 0.07 \) and \( \Delta \epsilon_{dyn}/\epsilon_{dyn} \approx 0.14) \) the obtained \( \epsilon_{dyn} \) throw the current measurements are almost identical with the values of \( \epsilon \) for the complex insulator measured at 1 \( MHz \). The refractive index is equal to \( [33] n = \sqrt{\epsilon_{ms}} = 1.43, 1.32, \) and \( 1.48 \) for Cur–B, –Fe, and –Cu, respectively (estimated accuracy \( \approx 0.1 \)), which is close to the refractive index measured optically at 1500 nm. These results confirm the suitability of application of RS mechanism to explain the experimental results.

In addition, for \( V_{ins} > 1.5 \) \( V \) (i.e. \( V_f > 2.4 \) \( V \)) the currents follow the mechanism of saturation induced by carrier trapping \( [34] \).

**Fig. 11.** Forward \( I(V_{ins}) \) dependence in accordance with RS model: \( \ln(I) \) vs. \( V_{ins}^{0.5} \) for MIS structures.

4.3. Optoelectronic properties

Under the illumination through glass plate of an incandescent-lamp light of maximum intensity at 630 nm and integral intensity of 266 \( W/m^2 = 183 \) \( \text{kLux} \), the \( I(V_f) \) characteristics of the samples were studied and the results are shown in Fig. 12. With illumination, the diode behaviour of the samples is constant and is enhanced, and the breakdown voltage \( (V_{bd}) \) decreases. However the forward current at \( V_f = 8 \) \( V \) was increased under illumination by 8.7, 3.9, and 10.1 times for Cur–B, –Fe, and –Cu, respectively. However, to compare the relative sensitivity of samples to the incident light, the integral optical responsivity \( (A/W) = [I_{ill} - I_{dark}]/[E_{ill}] \) is used, where \( I_{ill} \) and \( I_{dark} \) are respectively the currents under illumination and in dark, \( E_{ill} \) is the incident light energy, both currents were measured at \( -8 \) \( V \) gate voltage. The results of \( S^* = 0.57 \) \( A/W, 1.16 A/W, \) and \( 0.90 A/W \) for Cur–B, –Fe, and –Cu, respectively. Therefore, electrical (ac and dc) and optoelectronic properties of Cur–Fe are different from those of Cur–B or Cur–Cu, which may be due to the magnetic character of the coordinated Fe ions that can affect the properties of the complex and consequently affect the microstructure of the film sample. The photosensitivity of the samples can be explained by the photosensitivity of the Cur–M insulator so that they have different \( S^* \) values depending on the embedded metallic ion type. Thus, the production of a high density of free electrons with illumination the current flow mechanism is controlled.

5. Conclusions

Curcumin (Cur–M) coordinated with metal ions B, Fe, and Cu were synthesised and crystals produced. The optical and electrical properties of these Cur–M thin films were studied. Two absorption bands were observed in the visible range of all the investigated Cur–M. Also, it was found that the amorphous Cur–M films deposited on p-Si can be used and utilised to construct MIS structures within the usual device grade of fixed and interfaces charge density. Thus Cur–M films are good transparent insulators \( (p \approx 10^9 \) \( \Omega cm) \). The leakage current through Cur–M insulators obey Richardson–Schottky emission mechanism. The important find on Fig. 12 is the large optoelectronic sensitivity so that the electrical conductivity at 8 \( V \) increases under light illumination by \( \approx 400-1000\% \). In general, the systematic electrical study show that Cur–M films can be utilised for small-k \( (k \approx 2) \) production of microelectronic and optoelectronic devices.
References