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# **ORIGINAL ARTICLE**

# Two new Mn(II) coordination polymers: Photocatalytic property and treatment activity on colorectal cancer by inhibiting cancer cell migration and invasion

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#### **KEYWORDS**

Mn(II) compound; Photocatalysis; Colorectal cancer

act Τw ew binary Mn(II) coordination polymers, namely [Mn(5-MeO-ip)(DMF)]<sub>n</sub> (1) and (obb)]<sub>n</sub> (**2**) (.  $H_{2}O-H_{2}ip = 5$ -methoxyisophthalic acid,  $H_{2}Obb = 4,4'$ -oxydibenzoic acid), have thesized vin the self-assemble reactions of Mn(II) ions with two different dicarboxylic acid be optical band gaps for 1-2 are 3.21 eV, 3.50 eV, respectively, and their photocatalytic ligands. properties. the degradation of MV under UV light irradiation were also investigated. For the colrectal cancer treatment, the biological activity was evaluated and the specific mechanism was explored at the same time. Firstly, the inhibitory activity of the new compounds on the colorectal canviability was measured with CCK-8 assay. Next, the real time RT-PCR was used to measure cer activation of the VEGF signaling pathway in the colorectal cancer cells after compound treatment. © 2021 The Author(s). Published by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

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#### 1. Introduction

Colorectal cancer is one of the malignant tumors with a relatively high incidence. Its incidence rate is among the top five in the world. In the past, the incidence rate in China was relatively low (Haraldsdottir et al., 2014). According to the data released by our current National Cancer Center, the incidence and death rate of colorectal cancer in China are constantly rising. However, the molecular mechanism of the colorectal cancer is not completely clear. (Thanikachalam and Khan, 2019) Therefore, studying the molecular mechanism of the colorectal cancer may provide an experimental basis for the development

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of new molecular targeted therapies for the treatment of colorectal cancer.

In the last several decades, coordination polymers have attracted great interest, which primarily owing to the potential functional properties that make them can be used in the field of sensing, luminescence, gas storage, catalysis, magnetism, drug delivery, etc (Pan et al., 2019; Dutta et al., 2021; Liu et al., 2021; Pan et al., 2020; Li et al., 2020). A lot of research has proved that coordination geometries of metal ion, the structural characteristic, coordination sites of the organic blocks perform as crucial parts in in directing the formation of final structure of the coordination-bond driven self-assembly (Li et al., 2020; Fan et al., 2022). Thus, intelligent selection of structure-related organic blocks and metal ions with specific coordination geometries can help us to obtain the coordination polymers with desired structures and functional properties. Among the various types of organic ligands widely used in the syntheses of coordination polymers, multidentate carboxylate ligands serve as one of the most efficient organic ligands to construct coordination polymers, they display abundant coordination modes and possess high coordination capacities (Fan et al., 2021; Feng et al., 2017; Feng et al., 2015). These carboxylate-based coordination polymers usually exhibit exceptional thermostability and interesting physicochemical properties (Wang et al., 2021; Lu et al., 2021; Zhang et al., 2020). Most of such materials containing Cd(II), Co(II), Cu (II) or Mn(II) ions show promising applications as photocatalysts for the water purification (Li et al., 2021; Sun et al., 2015; Xiao et al., 2019).

Organic dye wastewater is seriously affecting human l due to the chemical stability and difficult biodegradabili of the organic dyes. In order to protect ecological balance d human health, it is urgent to develop new photo <sup>1</sup>vsts wi high photocatalytic activities for the photod adati of the organic dyes. Recently, there are various pes org ic-inorganic hybrid photocatalyst, coordination 1ym one type of them, has shown excellen notoca tic activities, lity (Kang good structure stability and rev al., 2020; Zhang et al., 2021). To a new coordination ain polymer-based photocataly we chose vo different dicarthis experim t, namely 5boxylic acid blocks methoxyisophthalic acid (5-MeCH<sub>2</sub>ip) and 4,4'-oxydibenzoic acid (H<sub>2</sub>obb), that can dopt using kinds of bridging or chelatal ions, assemble with Mn(II) ing modes linking with ions under solution al constition successfully, we obtained (II)-basid coord, from polymers formulated as  $(p)(D^{1}, f) = (1)$  and  $[Mn(obb)]_n$  (2). Compound two new M [Mn(5-M\_ip)(D) 1 has an exact d 3D structure, which has been proven by the structural a lyses of X-ray, and compound 2 exhibits an extended 2D layers structure. The thermostability, optical band gaps and photocatalytic activities of both compounds were investigated in this work. In the biological section, the application values of compounds 1 and 2 on the colorectal cancer was measured, followed by the mechanism exploration.

#### 2. Experimental

#### 2.1. Materials and instrumentation

All the raw materials including metal salts, organic ligands, organic solvent are commercially available from Jinan Hen-

ghua Company, and there is no need to do further purification. Using a Vario EL-Cube elemental analyzer to do elemental analyses of 1–2. Under the circumstances that a Rigaku Mini-Flex II diffractometer with Cu/K $\alpha$  radiation ( $\lambda = 1.54056$  Å) at the scan speed of 2°/minutes and one step size of 0.05°, the powder X-ray diffraction data of 1–2 were gathered. Thermogravimetric analyses were performed on a NETSCHZ STA-449C thermoanalyzer, which at a temperature range from 30°C to 800°C under a nitrogen atmosphere. The UV–Vis absorption spectra were collected on the Shimadzu UV–Vis 2501PC recording spectrophotometer.

# 2.2. Synthesis of [Mn(5-MeO-ip)(DMF)]n(1) and synthesis of [Mn(obb)]n(2)

Sealing the mixture of 5-MeO-1 bo (0.1 mmol DMF (2 mL), MnCl<sub>2</sub>·4H<sub>2</sub>O (0.1 mmol) ong wa H<sub>2</sub>O (1 1 L) in the glass vial of 20 ml, then framer heating it at a temperature of 110°C for a day and small. We in the temperature of the mixture was cooled to row terr erature paturally, colorless block crystals of **1** are gata and in 42°C yield, which relied on MnCl<sub>2</sub>·4H<sub>2</sub>G (Anal. Calcal %) for  $C_{12}H_{13}MnNO_6$  (322.17): C, 44.70°C, 4.5°N, 4.35. Found (%): C, 44.72; H, 4.16; N, 4.37.

ture of MnCu 4H<sub>2</sub>O (0.1 mmol), H<sub>2</sub>obb (0.1 mmol), http://dthefueline.com/states/sta

#### 2.3. X-ray crystallography

Choosing proper single crystals of 1–2, mounting it on a glass fiber under an optical microscope. The single crystal data of 1-2 was gathered on the Rigaku Mercury CCD diffractormeter, which is controlled by computer and equipped with graphitemonochromated Mo-K\alpha radiation ( $\lambda = 0.71073$  Å) at room temperature. The single crystal data reduction was performed by the CrysAlisPro, and the empirical absorption corrections were performed by the SADABS program (Sheldrick, 1996). Using the procedure of SHELXS-2014, via the direct method solve the structures of 1-2, and refine them via full matrix least-squares, which is based on  $F^2$  (Sheldrick, 2015). To refine all non-hydrogen atoms, anisotropic thermal parameters was performed, and adding all hydrogen atoms bonded to carbon geometrically, then using a riding model to refine isotropically. As we can see from the Table 1, there has summarized the detailed data of the crystallographic and structural refinements of the compounds. Chosen bond lengths (Å) and angles (°) of the compounds were given in Table S1.

#### 2.4. Photocatalytic experiments

Based on the previously reported literature (Yuan et al., 2020), the procedure of photocatalytic experiment conducted in this work is as follows: the finely grind samples of 1 or 2 (50 mg) was added into 100 mL methyl violet (MV) aqueous solution with the concentration of 10 mg/L, and the suspension was fur-

Sample	1	2
Formula	C <sub>12</sub> H <sub>13</sub> MnNO <sub>6</sub>	C14H8MnO5
Fw	322.17	311.14
Crystal system	orthorhombic	monoclinic
Space group	Pna2 <sub>1</sub>	<i>P</i> 2/c
a (Å)	7.437(3)	13.225(2)
b (Å)	15.164(7)	10.9116(14)
<i>c</i> (Å)	11.278(5)	4.8224(6)
α°	90	90
β°	90	95.118(10)
γ°	90	90
Volume (Å <sup>3</sup> )	1271.9(10)	693.13(17)
Ζ	4	2
Density (calculated)	1.683	1.491
Abs. coeff. $(mm^{-1})$	1.063	0.966
Total reflections	9331	5255
Unique reflections	2851	1564
Goodness of fit on $F^2$	1.003	1.104
Final R indices	R = 0.0234,	R = 0.0632,
$[I > 2 \operatorname{sigma}(I^2)]$	$wR_2 = 0.0460$	$wR_2 = 0.1772$
R (all data)	R = 0.0265,	R = 0.0754,
	$wR_2 = 0.0473$	$wR_2 = 0.1956$
CCDC	2,109,418	2,109,419

 Table 1
 The structure refinements and Crystal data of compounds 1–2.

ther stirred under the circumstances of darkness for half an hour to establish a balance between adsorption and desorption equilibrium. Then, a photocatalytic reaction was carried to through the XPA-7 type photochemical reactor, which is fitte with 100 W mercury lamp. At the given intervals, taking ou 5.0 mL mixture, separating it via centrifugation, and a physing it through UV–vis spectrometer subsequently under the same reaction environment, a blank test was also purformed with out any photocatalyst.

### 2.5. CCK-8 assay

The CCK-8 assay was condicted in this promit research to measure the inhibitory are vity of compounds r and 2 on the colorectal cancer viability This reformation was conducted nce of the instructions with only a little 226 cc rectale acer cells in the logical totally under the guidance change. In brief, growth phage pere collected and praced into the 96 well plates (5000 cells, cell). The cells were placed in the incubator of ∠ h. 48 mafter incubation, compounds 1 37℃, 5 %CO or and **2** were added nto the wells with  $0-80 \ \mu\text{M}$ . Then, the cell culture medium we discarded and the fresh medium was added containing 10 L CCK-8 reagent. After the indicated treatment, the absorbance of each well was measured at 450 mm. This experiment was repeated at least three times, and the results were presented as mean  $\pm$  SD.

## 2.6. Real time RT-PCR

The real time RT-PCR was conducted in this present research to measure the activation of the VEGF signaling pathway in the CT-26 colorectal cancer cells after compounds treatment. This conduction was finished strictly in accordance with the instructions. Shortly, the PC-1 pancreatic cancer cells in the logical growth phage were collected and seeded into the 6 well plates at the final destiny of  $10^6$  cells/ well. The cells were placed in the incubator of  $37^\circ$ C,  $5^{\circ}$ /<sub>6</sub>CO<sub>2</sub> for 12 h, then the new compounds were added for treatment with indicated concentrations. Next, the cells were collected and the total RNA in the cells were extracted with TRIZOL reagent. After measuring the concentration of the total RNA, which was then reverse transcripted into cDNA. The relative expression of the VEGF signaling pathway in the CT-26 colorectal cancer cells was measured with real time RT-PCR, the *gapdh* was used as the internal control gene. This experiment was repeated at least three times, and the results were presented as mean  $\pm$  SD.

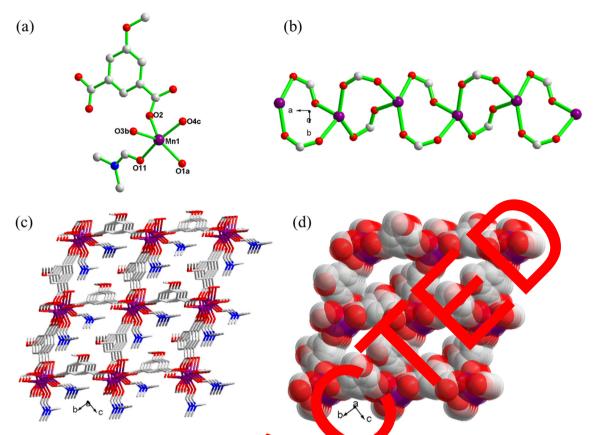
### 3. Results and discussion

## 3.1. Crystal structure of 1

As we can see from X-ray rystallog, phic analysis, the in the orthorhombic Pna2<sub>1</sub> space compound 1 crystallize group and is an extended 1D structure relied on 1D Mn-carboxylate crocks. In the a protectic unit of 1, there are one Mn(II) on, on 5-MeO-ip<sup>2</sup> agand and one terminal coordinated DMF molec. The Mn1 ion is five-coordinated by four anoxylate oxygen atoms from four different 5-MeO-ip<sup>2</sup> digands, and one oxygen atom from terminal coordinated F molecule, fording a trigonal bipyramid of {MnO<sub>5</sub>} D stances at the range of 2.115(2)-2.210(2) Å the Mn-O wi (Fig. The arboxylate group of each 5-MeO-ip2- ligand ere in unnorm bis-monodentate model. As shown in Fig. 1b, at {MnO<sub>5</sub>} polyhedrons are bis-bridged by two bisa nonodentate carboxylate groups, thus leading to the formation of a 1D Mn-carboxylate chain subunit extending along a axis, and in this 1D chain, the neighboring distance of Mn...Mn is 3.82 Å. Finally, these 1D chains are further linked by the 5-MeO-ip<sup>2-</sup> ligands to form an extended 3D structure for 1 (Fig. 1c). After remove the coordinated DMF molecules, it can be observed that the solvent-free framework for 1 contains 1D opened channels running along crystallographic a axis (Fig. 1d), and the solvent-accessible volume is 29.4% calculated by the PLATON program.

## 3.2. Crystal structure of 2

The crystal structure of **2** is an extended 2D layer crystallizing in the monoclinic P2/c space group with its asymmetric unit consisting of a half Mn(II) ion and a half obb<sup>2-</sup> ligand. Fig shows that, six carboxylate oxygen atoms formed the Mn1 ion, which locates in the center of a slightly distorted octahedron, these carboxylate oxygens are offered by six different obb<sup>2-</sup> blocks. The bond lengths of Mn-O are ranging from 2.121(3) to 2.297(3) Å. The V-shaped dicarboxylate ligand of obb<sup>2-</sup> adopt a  $(\kappa^1 - \mu_2) - (\kappa^1 - \mu_2) - \mu_6$  coordination mode with its two deprotonated carboxylate groups linking six different Mn(II) ions. It is worth noting that the connection between adjacent Mn ions is four-bridged by the carboxylate groups of obb<sup>2-</sup> ligands, which results in the formation of 1D Mn (II)-carboxylate chain running along crystallographic c axis (Fig. 2b). The 2-connected obb<sup>2-</sup> ligands further connected these 1D Mn(II)-carboxylate chains into an extended 2D layer (Fig. 2c). Viewing along crystallographic c axis, 1D opened



**Fig. 1** (a) Viewing of the coordination environment of Mn(1) ion in 1. (b) 170 An(II)-carboxylate chain structure. (c) The extended 3D structure of 1. (d) The 3D porous structure for 1 after renovments coordinated DMF molecules.

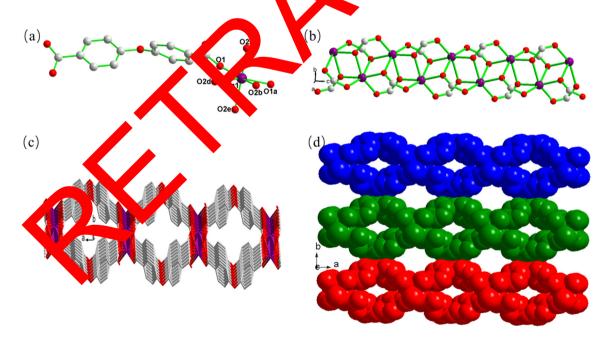


Fig. 2 (a) Viewing of the coordination environment of Mn(II) ion in 2. (b) The 1D Mn(II)-carboxylate chain structure in 2. (c) The extended 2D layered structure of 2 with 1D opened channels viewing along crystallographic *c* axis. (d) The layer-to-layer stacked 3D supramolecular framework under weak Van der Waals interactions.

channels in such 2D layer can be observed. Further, the final 3D supramolecular structure (Fig. 2d) was formed by the weak Van der Waals force-induced stacking of these 2D layers in -

AA- mode. As calculated by the PLATON program, the solvent-accessible volume for **2** is 121.7 Å<sup>3</sup>, corresponding to 17.6% of the unit cell volume.

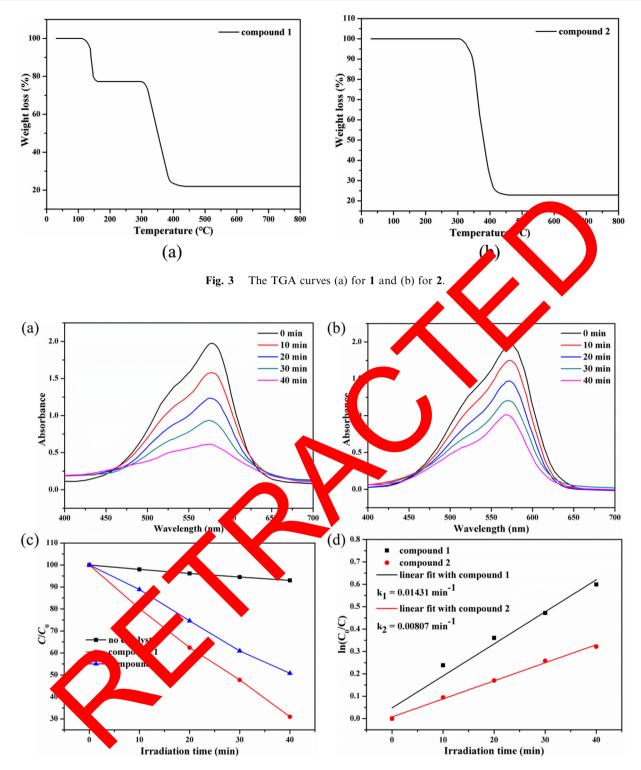


Fig. 4 (a) The UV–Vis absorption spectra of MV solution in the presence of 1 under different irradiation time. (b) The UV–Vis absorption spectra of MV solution in the presence of 2 under different irradiation time. (c) The plots of  $C/C_0$  versus irradiation time. (d) The first-order kinetic plots of photodegradation MV under the circumstances of the presence of compound 1 and compound 2.

#### 3.3. TGA and PXRD patterns

The experiments of PXRD were conducted to verify whether the as-synthesized samples of 1–2 are in pure phase. Fig shows that, S1, the PXRD patterns of 1–2 based on the synthesized samples fits nicely with their corresponding simulated patterns relied on the data of single crystal diffraction, clearly suggesting that the bulk samples of 1–2 are in single phase.

Furthermore, through the thermogravimetric analysis tests under  $N_2$  atmosphere (Fig. 3), their thermal stabilities were

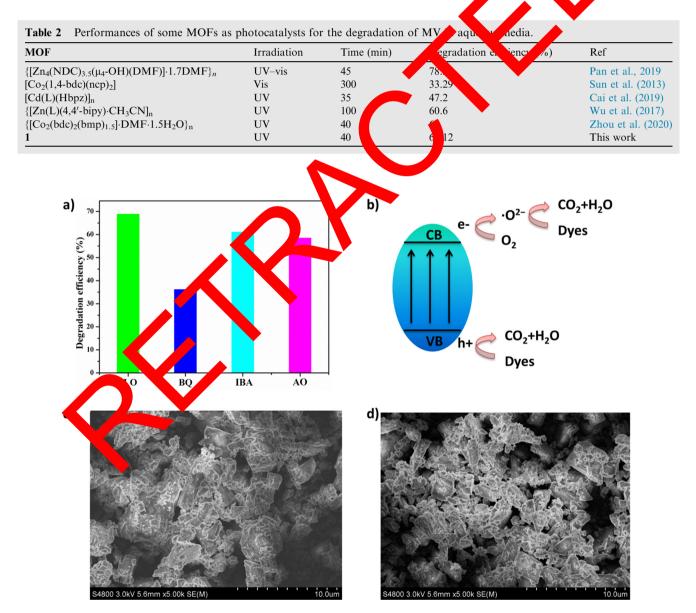
investigated at the same time. The significant two steps weightlessness were shown by the TGA curve of 1: one is appeared in the range of 112-160°C with a weight loss of 22.65% because of the evaporation of the coordinated DMF molecules (calcd: 22.69%), the other occurred in the temperature range of 295–417 °C, the decomposition of the organic ligand (calcd: 55.25%) lead to a weight loss of 55.36%. The TGA curve of **2** indicated that no observed weight loss took place before 306°C, and after that, the collapse of the framework was observed and weight loss continued to 458°C due to the loss of the organic ligand.

## 3.4. Band gaps and photocatalytic properties of 1-2

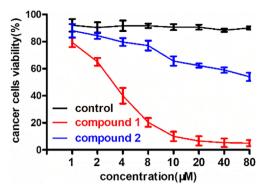
The UV–Vis absorption spectra of 1-2 as well as free organic ligands of 5-MeO-H<sub>2</sub>ip and H<sub>2</sub>obb were measured at room temperature (Fig. S2a). As calculated by the Kubelka-Munk

(K-M) function of  $F_{\rm R} = (1-R)^2/2R$  (*R*: the diffuse reflectance of the samples) based on the UV-vis absorption data, the band-gap energies ( $E_{\rm g}$ ) are 3.21 eV for 1 and 3.50 eV for 2 (Fig. S2b), indicating that such two compounds may be served as good photoactive materials in the field of photocatalysis.

Furthermore, the methyl violet (MV) was selected as a model dye contaminant to evaluate the photocatalytic activities of 1-2. And the blank experiment in the absence of photocatalyst was also performed under the same conditions. As shown in Fig. 4a and 4b, the absorption peaks of MV solution under the circumstances of the presence of compounds 1-2 declined gradually when exposing the UV light from 0 min to 40 min, and after 40 min, the degradation efficiencies of MV solution reached to 69.12%, 4 respectively, in the presence of 1-2 (Fig. 4c). The organization of much higher than previous liter ture MOFs f fficiency of 1 is MV degrada-(Table 2) However, the tion under the similar condition



**Fig. 5** (a) Photodegradation efficiency of MV solution in the presence of 1 along with different scavengers (BQ, IBA and AO). (b) The possible photodegradation mechanism of 1. The SEM diagrams of 1 before (c) and after (d) photodegradation experiments.

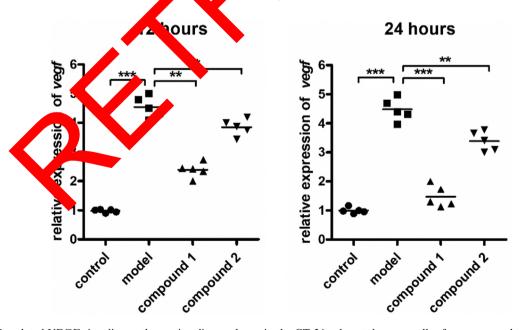


**Fig. 6** Significantly reduced viability of the CT-26 colorectal cancer cells after compound treatment. The CT-26 colorectal cancer cells in the logical growth phage were collected and seeded into the cell culture plate, then compounds **1** and **2** were added for treatment with serial different dilutions. The viability of the CT-26 colorectal cancer cells was measured with CCK-8 assay.

degradation efficiency of MV solution in the absence of photocatalyst reduced to 7.65% after 40 min UV light irradiation (Fig. 4c). This indicates that compounds 1–2 can be used as photocatalysts for the degradation of MV under UV light irradiation. It is noteworthy that the degradation efficiency of MV in the presence of 1 is higher than that in the presence of 2, suggesting that the photocatalytic performance of 1 is better than that of 2.

In addition, the degradation reaction rate constants with compounds as photocatalysts were also calculated using the model of pseudo-first-order kinetic:  $\ln(CO/C) = kt$  (C0: the intial concentration of MV, C: the concentration of MV at irradiation time t, k: the fitted first-order rate constant. As the can see from the Fig. 4b, there has a linear relation tap betworn the plots of  $\ln(CO/C)$  and t, and the linear fittings the the stants of 0.01431 min<sup>-1</sup> for 1, 0.00807 min<sup>-1</sup> for 2. The rate constant of 1 is higher than that of 2, which is in the same of the sequence of degradation efficiencies, further demonstrating that the better photocatalytic activity of 1 than 2.

The possible photocatalytic mechanism was also explored via the trapping experiments by selection of 1 as the representative (Zhao et al., 2019). Herein, various scavengers, such as benzoquinone (BQ) as a  $O_2^-$  scavenger, isopropanol (IBA) as a ·OH scavenger, ammonium oxalate (AO) as a h<sup>+</sup> scavenger, were used to explore the active species in the photodegradation reaction. When using the IBA and AO as the scavengers, the degradation efficiencies of MV were slightly decreased from 69.12% to 61.3%, 58.7%, respectively (Fig. 5). However, the degradation efficiency of MV was reduced from 69.12% to 36.4% using the BQ scalinger (Fig. a). This result indicates that  $O_2^-$  was the domined active speces during the photocatalytic process with this photocatalyst. I general, the mechanism of photocataly degradat, of dy using MOFs as catalysts is associated with servicenduc to neory. Previous studies have suggested that the electron charge transfer from the photoexcited aganic, and to the netal (LMCT) within MOFs account for their photoe aytic activity. Thus, the mechanismendy degradation of can be proposed as shown in Fig. 5b. When the irradiated with photons, electrons are n the valen band (VB) to the conduction band excited (CI , accompanied by Armation of positive-charged holes egradation of dye molecules will be mediated (h ) in the VB. idizing holes (h<sup>+</sup>). At the same time, superby he strongly d <sup>2–</sup>) formed by the reaction of electrons with radicals ( oxic have a strong ability to oxidize dye molecules. oxygen addition, we have also recorded the scanning electron crospe (SEM) diagrams of complex 1 before and after photocatalytic reactions, and it could be observed that its morphology does not obviously change, indicating the catalyst 1 does not decompose (Fig. 5c and Fig. 5d).



**Fig. 7** Regulated VEGF signaling pathway signaling pathway in the CT-26 colorectal cancer cells after compound treatment. The PC-1 pancreatic cancer cells in the logical growth phage were collected, then the compound was added for treatment at indicated different concentrations. The real time RT-PCR was recommended to measure the VEGF signaling pathway signaling pathway in the CT-26 colorectal cancer cells.

# 3.5. Compound significantly reduce the viability of the CT-26 colorectal cancer cells

After the design and synthesis of the new compounds 1 and 2 with novel structures, their treatment activity on the CT-26 colorectal cancer cells viability was determined. So, the CCK-8 assay was carried out in this research, and the viability of CT-26 colorectal cancer cells after compound treatment was measured. As the results showed in the Fig. 6, we can see there was a higher level of CT-26 colorectal cancer cells viability in the control group. After the treatment of compound 1, the CT-26 colorectal cancer cells viability was reduced significantly, which is significantly different from the control group, with P < 0.005. However, compound 2 only showed a little influence on the viability of the CT-26 colorectal cancer cells, which was much weaker than compound 1.

### 3.6. Compound reduced the VEGF signaling pathway activation in the CT-26 colorectal cancer cells

As we proved in the above experiment, the new compound showed excellent inhibitory activity on the viability of CT-26 colorectal cancer cells. As previously reported, the VEGF signaling pathway in the CT-26 colorectal cancer cells play an important role in the development of colorectal cancer cells. Thus, the real time RT-PCR was further conducted and the activation of the VEGF signaling pathway in the CT-26 colorectal cancer cells was measured. The results in Fig. 7 showed that there was a significantly increased level of VEGF signaling pathway in the CT-26 colorectal cancer cells compared in the normal cells. There was obviously difference between the two groups, with P < 0.005. Under the treatment of the n compound, the relative expression of the Y gnalin JL pathway in the CT-26 colorectal cancer cell was red ced sig nificantly, which is much more excellent the com

#### 4. Conclusions

In summary, two new Mp(I)-based condination polymers have been prepared base on two different carboxylic acid ent molecular structures of the dicar-show an extended 3D framework, ligands. Owing to diff boxylic acid ligands, an extended 3D framework, and 2 shows an extended ayered stacture. Moreover, both optic r band gaps of 2.31 eV and with . them have been acon 2.50 eV, ap the pho catalytic gradation efficiencies for 1–2 are 69.12 49.17 ectively, after 40 min UV light irradiation. The assay indicated that compound 1 was more excellent than a pound 2 on inhibiting the colorectal cancer cell viability. Next, be activation of the VEGF signaling pathway in the colorectal cancer cells was obviously reduced by compound 1, but not compound 2. Above all, we got this conclusion that compound 1 was much better than compound 2 on the colorectal cancer treatment through inhibiting the cancer cell viability, migration and invasion ability.

#### Data availability

Selected bond lengths (Å) and angles (<sup>°</sup>) for CPs 1-2 (Table S1); The PXRD patterns (a) for 1 and (b) for 2 (Fig. S1); (a) The UV-Vis absorption spectra for compounds

and free dicarboxylic acid ligands. (b) The diffuse reflectance spectra of K–M function versus energy for 1-2 (Fig. S2), the information could be found in the supporting information file.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Not applicable.

## Appendix A. Supplet stary aterial

Supplementation data to the particle can be found online at https://de.org/1016/j.araby/2021.103584.

#### R rences

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