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Synthesis, structure and antitumor studies of a novel decavanadate complex with a wavelike two-dimensional network

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ABSTRACT

A decavanadate salt $Na_4\{(HOCH_2CH_2)_3NH\}_2[V_{10}O_{28}]\cdot 6H_2O$ was synthesized *via* the reaction of sodium metavanadate and triethanolamine hydrochlorides in water solution, which exhibits an interesting wavelike 2D network in the crystal structure. The product also showed potent inhibitory activities against human laryngeal carcinoma epithelial cell line (Hep-2), human breast cancer cell line (MDA-MB-231) and hepatocellular liver carcinoma cell line ($HepG_2$), which was better than the positive control drug 5-fluorouracil (5-FU), implying that newly synthesized compound may be a potential candidate for the development of anti-cancer drugs.

1. Introduction

The supramolecular chemistry has attracted remarkable attention since Jean-Maire Lehn won the Nobel Prize in 1987. Now supramolecular chemistry has been widely used in molecular recognition [1–4], molecular self-assembly [5,6], catalysis [7,8] and molecular devices [9,10]. Supramolecular coordination chemistry, as an important component of supramolecular chemistry, plays an important role in self-assembly [11–13] and coordination supramolecular materials [14–16]. In supramolecular coordination chemistry, metal-coordination interaction is particularly interesting because metal-coordination bonds form highly directional and controllable manner [17–20]. Polyoxometalates (POMs) are a kind of inorganic clusters based on the assembly of MO_n (M = addenda atoms e.g. W^{VI}, Mo^{VI}, V^{V}) polyhedra of discrete anionic metal–oxygen clusters [21,22], which can be considered as an excellent receptor molecule and can be combined with organic ligands and metal ions to form supramolecular compounds [23,24]. As POMs have versatile optic, electric, magnetic properties and excellent catalytic and biological activities, the coordination supramolecular materials based on POMs can be expected to have a bright future. Thus, selecting suitable POM clusters and prepare novel organic–inorganic hybrid compounds constituting unique supramolecular architectures and topologies are great challenges for inorganic chemists.

Decavanadate $[H_nV_{10}O_{28}]^{(6-n)-}$ (n = 0–3) is an important class of POMs [25,26] which has been studied extensively in the past decades because of their excellent catalysis properties [27,28] and versatile bioactivities [29–32]. It is also a unique building block for constructing supramolecular assemblies as it can form hydrogen bonds with various organic ligands and the charges of the cluster can be adjusted by different protonation [33,34]. The supramolecular structures formed by decavanadates show various topological structures and also influence the thermal and electro properties of the polyanion. Therefore, it is of great significance to explore the supramolecular assemblies based on decavanadate, in which the selection of counterions for synthesis of coordination supermolecules is a key problem. There have been numerous reports of decavanadates with organic cations, metal cation or metal–organic coordinated cations [35,36]. However, the examples of decavanadates with a metal and an organic counterions are still rare.

In this work, we reported a novel of decavanadates complex with counterions of two different types: protonated triethanolamine as organic cations and sodium as metal cations. In particular, the triethanolamine cations and decavanadate were linked *via* coordination with sodium cations, resulting a stable two-dimensional reticular supramolecular assembly. The structure was fully characterized by single crystal X-ray diffraction, IR and ^1H NMR spectroscopy. The properties and thermal stabilities of the compound were investigated by UV–Vis spectroscopy, cyclic

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voltammetry and TG analysis. Furthermore, antitumor activity of the complex was also tested.

2. Experimental

2.1. Materials and general measurements

All chemicals, reagents, and solvents were of analytical grade and used without further purification. $Na_3(12H_2O)[H_3V_{10}O_{28}]\cdot 2H_2O$ (abbreviated as NaV10) and $[(n\text{-}C_4H_9)_4N]_3[V_{10}O_{28}H_3]$ (abbreviated as TBAV10) used for the comparation in antitumor test were prepared according to previous literature [37,38]. The IR (KBr pellet) spectra were recorded (400–4000 cm^{-1} region) on a Nicolet Magna 750 FT-IR spectrometer. UV spectrum was obtained on a Shimazu UV-250 spectrometer in the 200–400 nm range. Thermogravimetic (TG) measurements of the product have been performed using a METTLER TOLEDO apparatus in a temperature range of 25–800 °C with a scan rate of 10 °C/min in the presence of a nitrogen flow. Cyclic voltammogram (CV) were recorded on a CHI760E electrochemical work station, using a glassy-carbon electrode as working electrode, Pt electrode as counter electrode and Ag/AgCl electrode as reference electrode. The sweep rate is 25 $mV/s^{-1}$and sweep range is from −0.90 to 1.60 V. Cyclic voltammetric studies for complex were carried out in a water solution 1.0×10^{-4} M in complex and 0.1 M $NaClO_4$/0.01 M $HClO_4$ as supporting electrolyte.

2.2. Synthesis of $Na_4[\{(HOCH_2CH_2)_3NH\}_2][V_{10}O_{28}]\cdot 6H_2O$

The compound $Na_4\{(HOCH_2CH_2)_3NH\}_2[V_{10}O_{28}]\cdot 6H_2O$ (abbreviated as TEAV10) was synthesized by dissolving sodium metavanadate (2.42 g, 19.8491 mmol) and triethanolamine hydrochloride (0.6 g, 3.2319 mmol) into H_2O (20 ml) in a round-bottom flask. The solution was adjusted to a pH of 3.5 by adding HCl dropwisely and then heated at 65 °C for 48 h. After the reaction the mixture was filtered and the yellow-green filtration evaporated slowly in the open air at room temperature. Yellow crystals deposited from the filtration after 1 week with 30% yield.

2.3. X-Ray crystallography

The single crystal X-ray diffraction in this work were made on a Bruker P4 diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The raw frame data were processed by SAINT version 7.68A to yield the reflection data. Subsequent calculations were carried out using OLEX-2 program [39]. The structure were solved by direct methods and refinements were performed using full-matrix least-squares technique. The structure was refined as a 2-component twin and the twin law was detected using PLATON version 1.17 [40].

2.4. Anti-proliferative activities

The anti-proliferative activities were performed on hepatocellular liver carcinoma cell line ($HepG_2$), human cervical cancer cell line (Hela), human laryngeal carcinoma epithelial cell line (Hep-2) and human breast cancer cell line (MDA-MB-231). The cells were cultured in Dulbecco's modified Eagle's medium (DMEM; Gibico) supplemented with 10% fetal bovine serum (FBS), 100 U/mL penicillin G and streptomycin (Sigma, USA). After 3–4 days, exponentially growing cells were seeded in 96-well plates and grown to subconfluence. The growth medium was removed and the cells were incubated with various concentrations of tested compounds, 5-Fluorouracil (5-FU) was used as a positive control. Plates were incubated at 37 °C in a humidified atmosphere containing 5% CO_2 for 48 h. The MTT assay was used to determine cell viability as an indicator for cell sensitivity to the complexes [41]. Briefly, the cells were incubated with MTT (5 mg/mL in PBS) during 4 h at 37 °C. The formazan products were dissolved in DMSO and determined by the absorbance at 492 nm. All data were analyzed with SPSS software, and results were expressed as IC_{50}, defined as the concentration causing a 50% reduction or inhibition of cell viability.

2.5. Flow cytometry analysis of cell death

$HepG_2$, Hep-2 and MDA-MB-231cells were seeded in 6-well plate format at 100,000 cells/ well using 2 mL DMEM media supplemented. The cells were exposed for 48 h to TEAV10. Then cells from each individual well were centrifuged at 900 rpm for 5 min, washed two times with cold PBS and then resuspended in 500 μL of binding buffer. Each cell solution was added 5 μL of Annexin V-FITC and 5 μL of PI. After incubation for 20 min in the dark, cell suspensions were gently homogenized, and analyzed by flow cytometry.

3. Results and discussion

3.1. Synthesis and spectroscopy

Although decavanadate salts have been widely studied in the past decades, the counterions of the decavanadates were usually metal, coordinated ions and substituted ammonium cations. To explore more particular assemblies in this system, we introduced TEA as an organic chelating cation into the decavanadate salts together with sodium ions. The compound TEAV10 were synthesized by a simple flask heating reaction of a water solution containing sodium metavanadate and TEA hydrochloride. The ratio of the reactant should be precise as shown in the experiment section. The crystal of the product cannot be obtained without heating, indicating that a proper heating may help the complex form a stable structure which is common in coordinate chemistry.

Fig. S1 showed the IR spectrum of compound. In low wavenumber region, absorption bands at 968, 946, 817, 747, 600 cm^{-1} were attributed to ν_s (V=O); ν_{as} (V—O—V) and ν_s (V—O—V) in the decavanadate cluster, respectively [42]. The medium bands at 1093, 1058 and 1031 cm^{-1} were assigned to the ν(C—O) and ν(C—N) in triethanolamine. The ^1H NMR spectrum of the compound exhibited two triplet peaks at 3.44 and 3.90 ppm which were assigned to the methylene hydrogens in the TEA cation. These results proved that TEA has been successfully incorporated into the decavanadate salt.

3.2. Crystal structure

The structure of the compound was shown in Fig. 1. The compound crystalized in monoclinic system $P2_1/n$. The unit cell was consisted of two polyanions, four TEA cations, eight sodium ions and twelve water molecules. The polyanion represents the typical structure of the decavanadate cluster. The distances between vanadium and the doubly bridging oxygen are in range of 1.81–1.85 Å while the bond lengths of the vanadium and the triply bridging oxygen are nearly 2.00 Å, which is in agreement with the cluster anion $[V_{10}O_{28}]^{6-}$ in previous reports [36]. Each of TEA ligand was chelated to two sodium cations, in which one of the three hydroxyl groups coordinated with two sodium ions while the other two were monodentate. The C—N—C bond angles in the TEA ligands were 110.74°, 112.11° and 115.61°, which is larger than the non-protonated TEA molecules, indicating that TEA ligands in this structure were protonated. The sodium cations all adopt octahedral six-coordinated configuration which chelated to a terminal

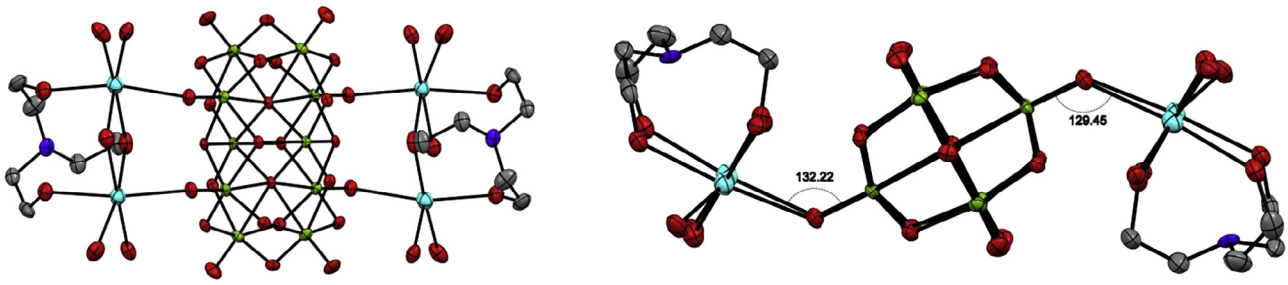

Fig. 1. ORTEP drawings of the molecular structure of TEAV10 viewed along *a* axis (left) and *ac* diagonal (right). Green sphere: V; teal sphere: Na; red sphere: O; blue sphere: N; grey Sphere: C. (Color online.)

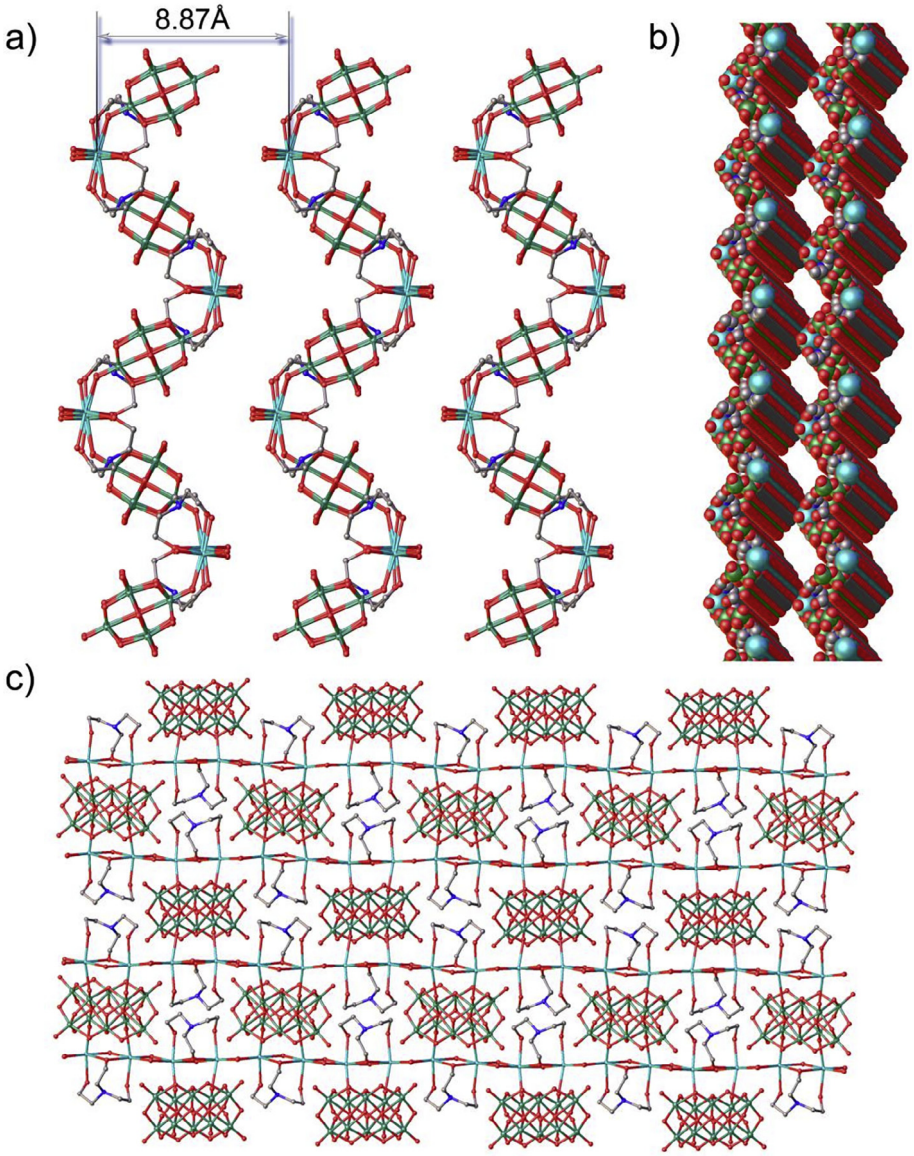

Fig. 2. (a) Packing diagrams of TEAV10 along *ac* diagonal; (b) spacefill presentation of TEAV10 along *ac* diagonal; (c) packing diagrams of TEAV10 along *a* axis. Hydrogen atoms were omitted for clarity.

oxygen(O_t) from the decavanadate cluster, two oxygens from the hydroxyl groups of the triethanolamine ligand and three water oxygens. The Na-O distances are in the range of 2.40–2.50 Å. Each of the sodium ion were connected to its two neighbors by hydroxyl and water oxygens, forming a 1D chain along the *ac* diagonal line. Furthermore, the sodium chains were linked by decavanadate

Table 1
Antitumor activities of the compounds against four human tumor cells.

Compound	In vitro cytotoxicity IC_{50}[b] (μg/mL)			
	Hela[a]	Hep-2[a]	MDA-MB-231[a]	$HepG_2$[a]
TEAV10	53.1 ± 12.1	13.5 ± 6.4	2.9 ± 0.81	16.4 ± 3.0
NaV10	68 ± 18.4	10.4 ± 4.6	1.2 ± 0.47	17.7 ± 6.7
TBAV10	57.4 ± 19.9	10.5 ± 4.2	1.6 ± 0.34	76.7 ± 7.6
5-FU[c]	30.4 ± 6.1	20.8 ± 6.9	18.3 + 6.4	27.2 ± 3.9

[a] *Abbreviations:* Hela—Human cervical cancer cell line; Hep-2 — Human laryngeal carcinoma epithelial cell line; MDA-MB-231—Human breast cancer cell line;$HepG_2$ — Hepatocellularliver carcinoma cell line.
[b] IC_{50} — Compound concentration required to inhibit tumor cell proliferation by 50%.
[c] 5-Fluorouracil, used as a positive control.

cluster through the terminal oxygen coordination, resulting in a 2D frame structure. Interestingly, the angles of the Na—O_t—V were 129.45° and 132.22°, therefore the 2D assembly is not complanate but an undulant wavelike structure. The distance between two neighbor wavelike planes is 8.87 Å. To the best of our knowledge, this kind of assembly is rather peculiar in POM systems (see Fig. 2).

3.3. Thermal stability

The TG curve of TEAV10 shown in Fig. S2 indicates a three-stage weight loss. The first weight loss between 139 °C and 159 °C corresponds to the loss of six coordinated water molecules, which was in accord with the single-crystal XRD analysis (exp.:7.59%, theo.: 7.47%). The second weight loss between 159 °C and 379 °C can be assigned to the loss of two TEA cations (exp.: 20.51%, theo.: 20.61%). A further weight loss occurs from 379 °C and continues up to 800 °C, in which decavanadate clusters were slowly collapsed and decomposed into vanadium oxides.

3.4. Pharmacology evaluation

3.4.1. The in vitro cytotoxic effects of compounds on cancer cells

In order to examine whether TEA cations have positive influence on cytotoxic effects of decavanadates, we compared the two classical non-supramolecular structures of decavanadate (NaV10, TBAV10) on proliferation of Hela, Hep-2, MDA-MB-231 and $HepG_2$ cell lines by the standard MTT (3-(4,5-dimethylth- iazol-2-yl)-2,5-diphenyltetrazolium bromide) assay using 5-FU (5-Fluorouracil) as

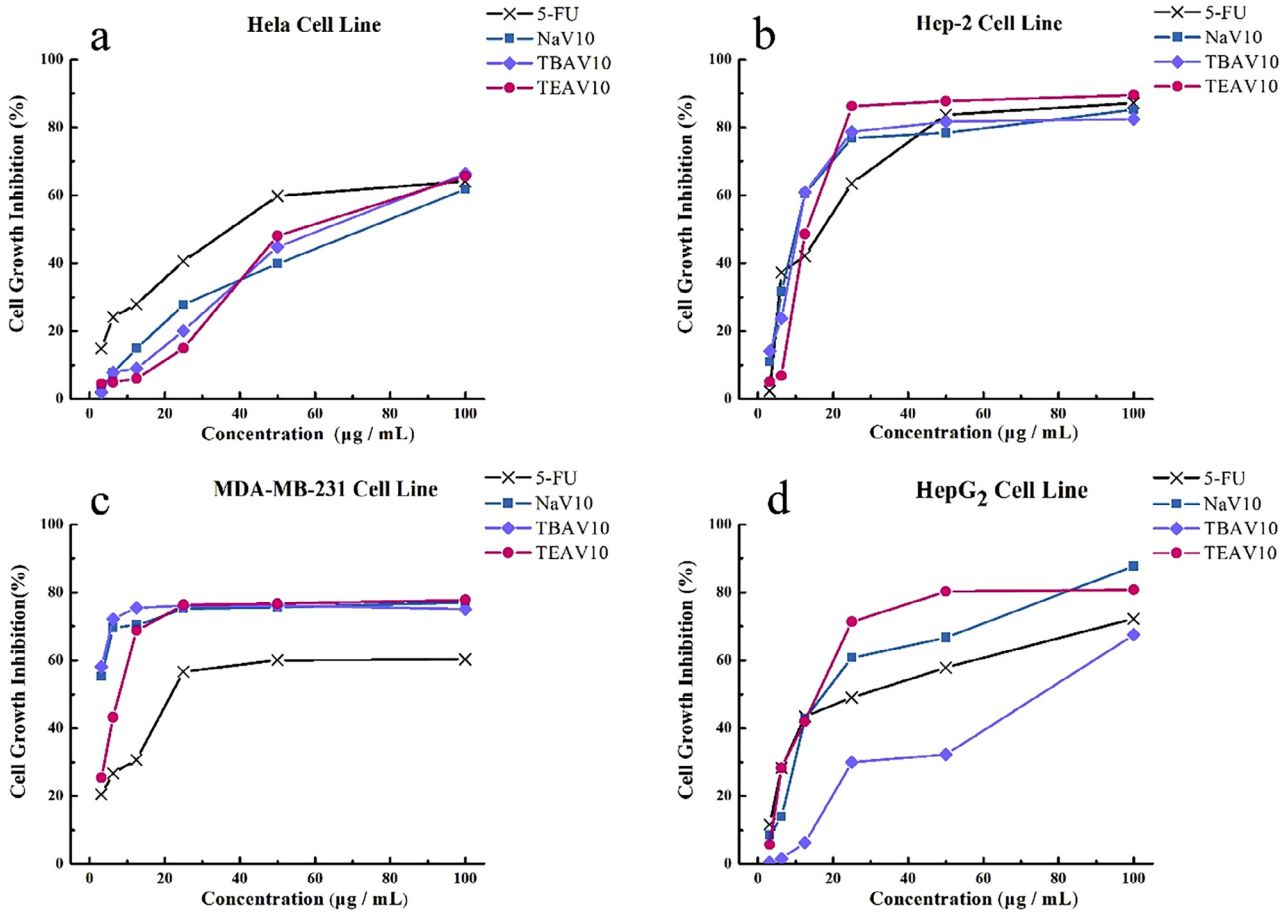

Fig. 3. Dose–response analysis of cell growth inhibition activity for TEAV10, NaV10, TBAV10 and 5-Fu against Hela cells (a), Hep-2 cells (b), MDA-MB-231 cells (c) and $HepG_2$ cells (d).

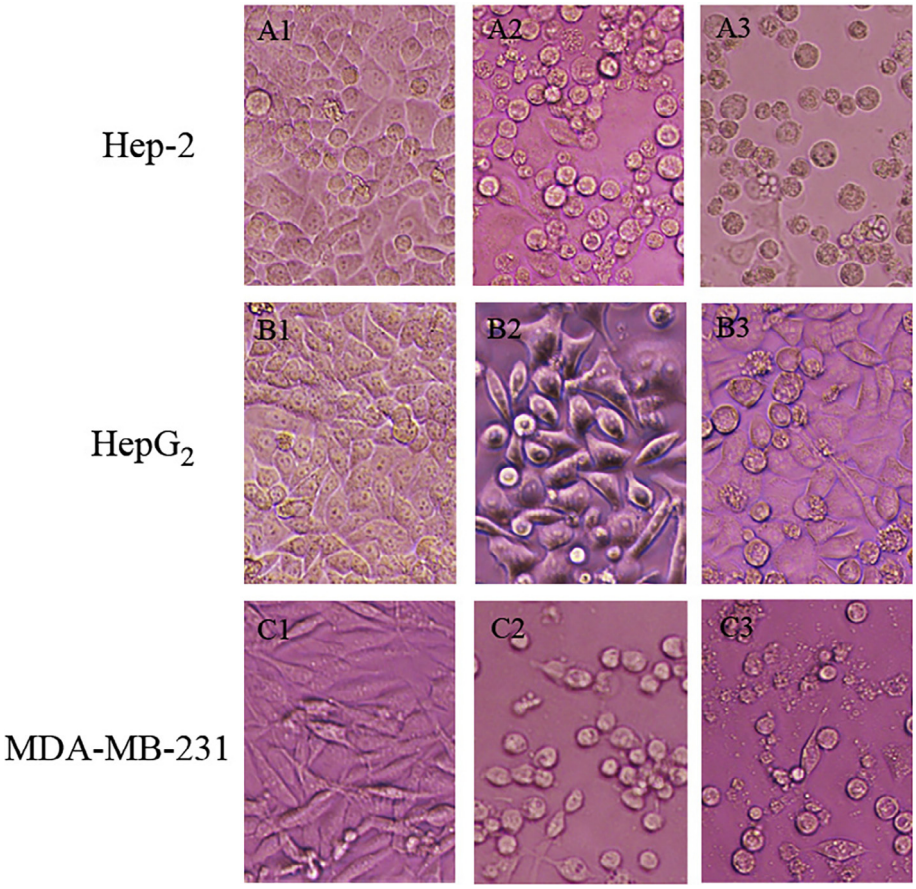

Fig. 4. Morphology image of Hep-2, HepG2 and MDA-MB-231 cells treated with compound TEAV10. The Hep-2, HepG2 and MDA-MB-231 cells were left untreated or treated with specific concentrations of TEAV10 for 48 h pi at 37 °C, respectively, the inhibitory effects against the proliferation of tumor cells were observed with respect to cellular morphology. A1: Hep-2 cell control, A2: 20 μg/mL TEAV10, A3: 40 μg/mL TEAV10; B1: HepG2 cell control, B2: 20 μg/mL TEAV10, B3: 40 μg/mL TEAV10; C1: MDA-MB-231 cell control, C2: 6.25 μg/mL TEAV10, C3: 20 μg/mL TEAV10.

a positive control. The IC_{50} values were calculated and are listed in Table 1. It is apparent from Table 1 that newly synthesized compound TEAV10, two classical analogue NaV10 and TBAV10 are active against the tested cell lines and have almost the same strengths. They showed the most potent cytotoxic effects against MDA-MB-231, with IC_{50} values of 2.9, 1.2 and 1.6 μg/mL. They also displayed remarkable activities against Hep-2 and $HepG_2$ cells except TBAV10 was only slightly active against $HepG_2$ cells. All compounds exhibited a higher inhibitory activity than the commercial 5-FU under the same conditions (except TBAV10 against $HepG_2$ cells). By contrast, these compounds presented globally lower activities in Hela cell lines. Previous study has demonstrated that NaV10 showed an obvious inhibitory effect against the proliferation of tumor SMMC-7721 and SK-OV-3 cell lines [43], which is consistent with our finds. However, antitumor effect of compound TBAV10 has not been reported to date. Here, we also demonstrated newly synthesized compound TEAV10 possess potent cytotoxicity against several tumor cells. These studies illustrated that the introduction of organic ammonium cations did not have a positive effect for the inhibitory activities of the decavanadates and these compounds might be considered as promising lead scaffold for further design and synthesis of potential anticancer agents.

Furthermore, the concentration-dependent inhibitory effects on cell proliferation for all tested compounds have been displayed in Fig. 3. The results indicated that all compounds exhibited significantly cytotoxic effects on Hep-2 cells, with inhibitory rates ranging from 82.5% to 89.6%. TEAV10, NaV10 and TBAV10 also showed high inhibitory activity against MDA-MB-231 cells, however, a low level of inhibition of 5-FU was observed. TEAV10 and NaV10 were more potent than TBAV10 and 5-FU against $HepG_2$ cells lines. Especially, these compounds could exhibit inhibitory effects against Hela cells lines only at high concentrations, with the lowest inhibition rate.

Changes in cell morphology can be sign of cytotoxicity of the compounds. The selective morphological image of the cells treated with newly synthesized compound TEAV10 at specific concentration for 48 h are shown in Fig. 4. The mock cells showed a regular shape, clear boundaries and good adherence, and the compound-treated cells appeared a rounded-up appearance and detached from the dish at the lower concentrations, while a nearly complete inhibition was observed at the higher concentration.

Apoptosis is the final effect of the treatment with multiple chemotherapeutical agents in cancer. Many anticancer drugs (as cisplatin) are effective against cancerous cells by causing apoptosis by the generation of reactive oxygen species (ROS) and DNA damage [44,45]. Apoptosis is a programmed mode of cell death that is accompanied by numerous morphological as well as biochemical changes in the cellular architecture. In the assays, flow cytometry was performed to investigate the effect of TEAV10 on cellular apoptosis or death. In the assays, the cells were stained with Annexin-V-fluorescein and propidium iodide, fluorescence drifting indicates cells undergoing apoptosis. The cells treated with TEAV10 showed a significant fluorescence drift to the right (representative of early apoptosis) and to the upper-right quadrant (representative

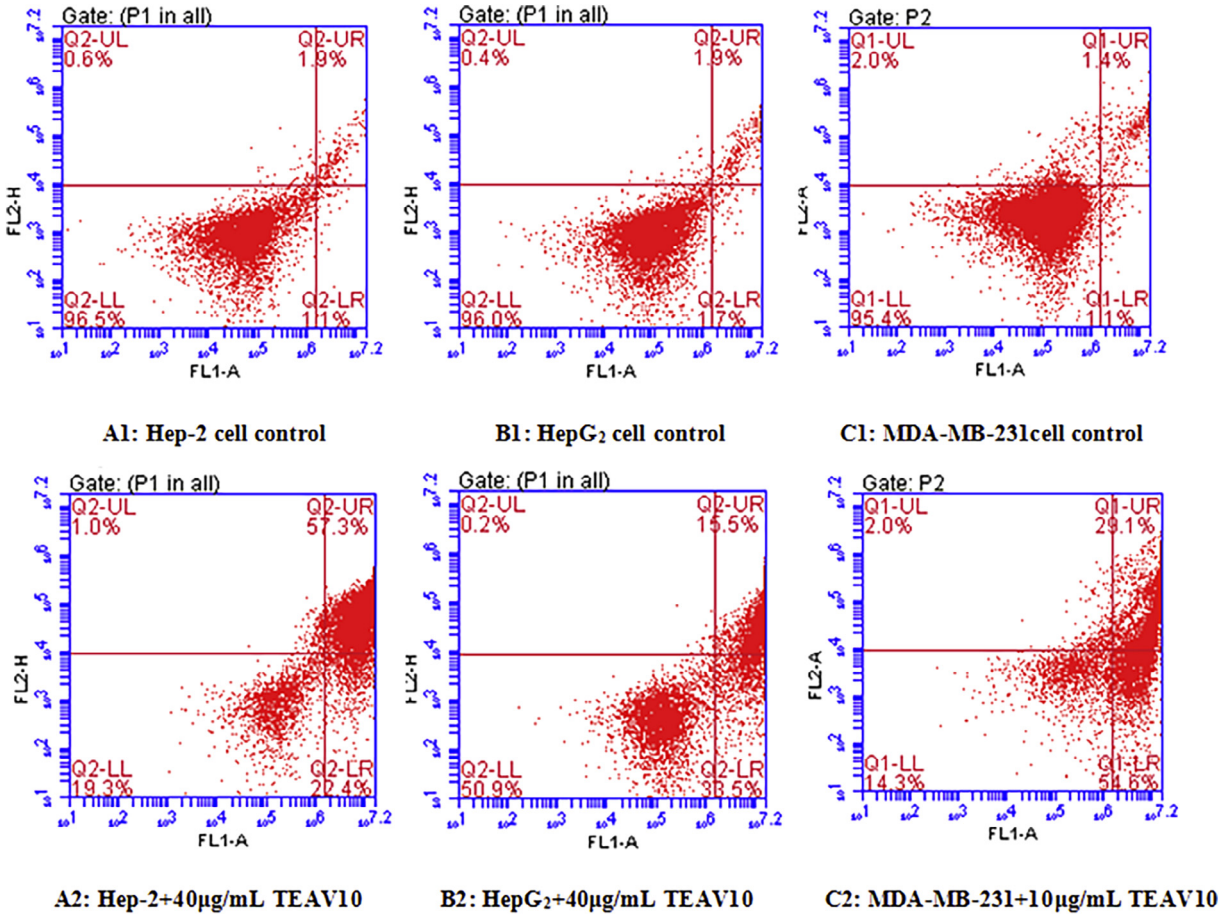

Fig. 5. Inhibitory effects of compound TEAV10 on apoptosis or death of Hep-2, HepG2 and MDA-MB-231. The Hep-2, HepG2 and MDA-MB-231cells were left untreated or treated with specific concentrations of TEAV10 for 48 h, the cells were stained with Annexin-V-fluorescein and propidium iodide and measured using flow cytometry.

of late apoptosis or death) (Fig. 5. A2, B2, C2), while fluorescence drifting could hardly be observed in control cells (Fig. 5. A1, B1, C1), which indicates compound TEAV10 could potently induce apoptosis or death of tumor cells.

4. Conclusion

In this work, we reported an interesting decavanadate complex with organic chelating cations and sodium cations, which is synthesized by a simple flask heating reaction. In the crystal structure of the compound, the decavanadate cluster and the triethanolamine cations were coordinated with sodium ions, forming an interesting wavelike 2D assembly in the crystal structure, which is very rare in the reported decavanadate salts. The compound also exhibits strong antitumor activities against several tumor cells, implying that it may be a potential candidate for the development of anti-cancer drugs.

Acknowledgments

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Appendix A. Supplementary data

CCDC 1845670 contains the supplementary crystallographic data for TEAV10. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.poly.2018.08.052.

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