Original Article

A facile method for synthesis of nanostructured copper (II) oxide by coprecipitation

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ABSTRACT

Due to their versatility e wide range of physical and chemical properties, transition metal oxides are very promising as nanostructured materials. Copper oxides are indicated for applications in increasingly wider areas as gas sensing, catalysts, solar cells, electrochromic devices, and antimicrobial materials. In this study, copper (II) oxide nanoparticles were synthesized by the coprecipitation method from two precursors: copper sulfate and copper chloride. The evaluated precursor concentrations were 0.1 M and 0.2 M and temperatures were 50 °C and 75 °C. Different results were found, according to synthesis parameters and precursor used. Using copper sulfate as a precursor, a single phase of polycrystalline CuO could be obtained, and only a mixture of crystalline phases of CuO and clinoatacamite (Cu2(OH)3Cl) were found using copper chloride as the precursor. A known polymorphism phenomenon was observed in clinoatacamite according to synthesis reaction parameters, and all three polymorphs were found mixed with CuO nanoparticles. Crystallite sizes of about 10 nm were found in this study, with leaf and rod-like nanostructured particles with dimensions range from 190 to 700 nm, and very good thermal stability. Under the synthesis conditions selected in this study, nanostructured materials made with copper sulfate precursor exhibited smaller crystallite sizes and better thermal stability. The coprecipitation method used was considered adequate to obtain nanostructured materials, with the advantages of being simple, fast and inexpensive.

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1. Introduction

The broad properties spectrum of nanostructured transition metal oxides has received great attention from researchers. However, their size and morphology control is still in development [1]. And, depends on the reaction conditions and several interdependent parameters such as temperature, electrostatic interactions and sternal confinements [2].

Copper Oxide (CuO) due to its excellent physical and chemical properties [3] has numerous potential applications, including research fields as catalysis [4,28] and gas sensors [5,31]. More advanced applications as solar cells, light-emitting diodes, photocatalyst, electrochemical, elec-
trochromic devices are possible research fields for copper oxide nanostructured materials [6]. Furthermore, nanostructured CuO is used for several applications in the removal of inorganic pollutants and could be used as potential components for the fabrication of nanodevices [7]. Nanoparticles specially designed for antimicrobial applications in biotechnology and pharmaceuticals have great demand and its market was valued at US$ 17.5 billion in 2011 [8].

Copper is widely used as a potent biocidal for centuries and both gram-positive and gram-negative bacteria are killed when exposed to high copper concentrations [32]. The same biocidal effect is reported in the literature for copper oxide nanoparticles as a promising material to prepare antimicrobial textiles, with very effective results in cotton fabrics [9]. Recent developments have also reported the antimicrobial properties of copper oxide nanoparticles in textiles for biomedical applications [10]. The copper oxide impregnated into cotton or polyester fabrics has shown good biocidal results against various microorganisms and viruses. The low risk of causing unfavorable effects on the skin is an advantage of copper oxide nanoparticles application [11].

Nanostructured materials have a high antimicrobial effect due to its high surface area to volume ratio and the dependence of its cytotoxicity to particle size is reported in the literature. It was also observed higher levels of cytotoxicity in copper oxide nanoparticles than other transition metal oxides such as iron oxide, titanium dioxide, and zinc oxide [12,29].

Their promising properties and low cost compared to other metal oxides have increased the interest in the development of nanostructured materials of copper (II) oxide. Its structure is considered more efficient and stable for antimicrobial applications than copper (I) oxide at room temperature [3].

Many synthesis routes were reported to prepare nanostructured CuO particles, each one with advantages and drawbacks, including sol-gel [13], hydrothermal [14], thermal decomposition [15,16], microwave irradiation [17], alchemical [18] and sonochemical processes [19,30]. In this context, this study proposed the synthesis and characterization of copper (II) oxide nanoparticles using two different precursors. The coprecipitation synthesis method was chosen for its simplicity, reaction rates at low temperatures and low cost.

Copper oxide nanoparticles were produced in two reaction conditions, in an alkaline medium using sodium hydroxide (NaOH). The precursors were copper sulfate (CuSO₄·5H₂O) and copper chloride (CuCl₂·2H₂O). The synthesis was conducted at temperatures of 50°C and 75°C and the concentrations of metal salts were 0.1 M and 0.2 M. After preparing, copper (II) nanoparticles made with copper sulfate as precursor showed better results (minor crystallite sizes). The coprecipitation method was considered suitable for obtaining nanostructured materials, with the advantages of being simple, fast and inexpensive.

2. Experimental

2.1. Materials

Two copper salts were used to prepare CuO nanostructured particles, copper sulfate (CuSO₄·5H₂O) 98 % PA (SYNTH) and copper chloride (CuCl₂·2H₂O) 99 % PA (VETEC); sodium hydroxide (NaOH) 97 % PA (NAFAN) was used as an alkaline reagent. All reagents were used without purification and solutions prepared with distilled and deionized water.

2.2. Synthesis method

Copper oxide nanoparticles were prepared using a chemical coprecipitation method and NaOH as an alkaline reagent. First, 100 mL precursor solutions were prepared using distilled and deionized water (0.1 and 0.2 M) and kept in constant magnetic stirring in a rounded bottom three-neck flask. The temperature was raised and maintained at 50 or 75°C as summarized in Table 1.

After, an alkali (NaOH 1.0 M) solution previously prepared, was added drop-wise using a burette, until a volume ratio of 1:1 was obtained. Constant stirring (500 rpm) was maintained in the whole experiment and alkali solution was added at a flow rate of 5 mL/min obtaining a pH 14 at the end of alkali addition. The solution color gradually changed from blue to black, and a considerable amount of black precipitate was formed. The reaction conditions were maintained for an additional 30 min at the same temperature and constant stirring. The precipitates obtained were filtered under vacuum and washed with deionized water at room temperature. After washing, the powder was dried in a vacuum drying oven for 5 h at 80°C, and characterization was performed.

2.3. Characterization of CuO nanoparticles

The samples were characterized by X-ray diffraction (XRD) analysis to evaluate the existing crystalline phases. The equipment used was a Philips diffractometer X’pert model with copper Kα radiation (λ = 1.5418 Å), 40 kV and 30 mA, 2θ ranging from 5 to 110 and an angular step of 0.05°.

Thermogravimetric analysis was applied (TGA) for measuring the mass variation according to temperature. The exothermic and endothermic peaks were also analyzed by differential scanning calorimetry techniques (DSC). A thermal analyzer Netzsch Simultaneous Thermal Analyzer STA 449 F3 was used and samples were evaluated in the range 22–1000°C at a heating rate of 10°C/min, platinum sample holder and with flowing nitrogen atmosphere of 20 mL/min.

The morphology of the material was analyzed at the Electronic Microscopy Laboratory (LCME) at UFSC, through scanning electron microscopy techniques (SEM) and transmission electron microscopy (TEM). For SEM a JEOL model...
JSM-6390LV was used, coupled to EDX (Energy Dispersive X-ray Spectroscopy), which was used for qualitative elemental analysis. Samples were pre-coated with gold and the images were obtained at 15 kV accelerating voltage. The TEM analyses were performed in a transmission electron microscope JEOL JEM-1011 model, with 100 kV voltage.

3. Results and discussion

3.1. X-ray diffraction of nanoparticles synthesized from copper sulfate

The XRD patterns of prepared precipitates with copper sulfate as a precursor are presented in Fig. 1. The characteristic peaks in the XRD patterns are shown $2\theta = 35.6^\circ$; $2\theta = 38.7^\circ$; $2\theta = 48.9^\circ$; corresponding to planes (002) and (111); (200) and (111); and (202) of CuO. With monoclinic crystal system, space group C2/c and lattice parameters: $a = 4.668\ \AA$, $b = 3.423\ \AA$, $c = 5.132\ \AA$, $\alpha = 90^\circ$ and $\beta = 99.54^\circ$ [20]. Similar results were found by Lange et al. [33], using the coprecipitation method with a 0.02 M solution of copper acetate as a precursor. Comparable XRD results were found using copper sulfate as a precursor and the coprecipitation method for coating cotton fibers [9]; and using coprecipitation method with subsequent application of ultrasound [21].

In sample C, additional peaks observed are due to the formation of brochantite (Cu$_4$(SO$_4$)(OH)$_6$) de posnjakite (Cu$_4$(SO$_4$)(OH)$_6$H$_2$O), confirmed by the XRD patterns presented in Fig. 2. In the work of Darezereshki and Bakhitiari [22] similar results were obtained using CuSO$_4$ as precursor salt.

According to Frost [23], in highly alkaline medium, copper (II) oxide is the most stable form and brochantite becomes the more stable phase in case of pH in 4–6 range.

All samples exhibited similar diffraction patterns with small variations in intensity and width of the peaks. Only when prepared at 75°C and at 0.1M CuSO$_4$ the presence of compounds like brochantite and posnjakite were detected indicating that the increase in temperature and the use of lower concentrations difficult the removal of sulfate ions (SO$_4^{2-}$) during the formation of CuO crystals. The absence of impurity peaks, like Cu(OH)$_2$ and Cu$_2$O, demonstrated the high purity of the nanostructured material produced.

3.2. X-ray diffraction of nanoparticles synthesized from copper chloride

In the diffraction data obtained for the precursor copper chloride, there was the formation of different crystalline phases (Fig. 2). Copper oxide characteristic peaks were found at $2\theta = 35.5^\circ$; $2\theta = 38.7^\circ$; $2\theta = 48.9^\circ$, confirming the formation of copper oxide in the nanostructured particles.

However, other peaks were found, suggesting a mixture of crystalline phases. Peaks at: $2\theta = 16.3^\circ$; $2\theta = 32.5^\circ$; and $2\theta = 39.7^\circ$; $2\theta = 53.8^\circ$; indicated the formation of chlorinated compounds such as clinoatacamite (Cu$_2$(OH)$_2$Cl) and its polymorphs.

According to Frost [23], the mineral clinoatacamite (monoclinic) has polymorphism and also is shown in the form of botallackite (monoclinic) and atacamite (orthorhombic). The stability order for these phases is clinoatacamite > atacamite > botallackite and, according to the Ostwald rule in a chemical reaction where different products are formed, the first being formed is preferably the lowest Gibbs free energy chemical compound, botallackite, in this case.

It was observed in the results, that increasing temperature or the precursor salt concentration favored the formation of chlorinated compounds. Under the synthesis conditions in this study, the formation of a single phase of copper oxide was not observed.

As prepared, CuO nanostructured particles made with copper sulfate precursor demonstrated better purity and could be used in applications like antimicrobial textile materials without further purification. Using copper chloride as precursor salt, chlorinated impurities were found.
3.3. Average crystallite size

The crystallite size, $D$, was estimated according to the Scherrer equation (Eq. 1), using as reference the highest intensity peak at $2\theta = 35.6^\circ$. The angle parameter ($\theta$) and full width at half maximum (FWHM) were calculated using the OriginPro 8 software, $\lambda$ is the copper K$_\alpha$ wavelength 1.5418 Å and the results are shown in Table 2.

$$D = \frac{0.9\lambda}{FWHM \cdot \cos \theta}$$  \hspace{1cm} (1)

Smaller nanoparticles were obtained using CuSO$_4$ as precursor salt. The reaction temperature and concentration of precursor salt also influenced the crystallite size, where an increase of temperature generates an increase of crystallite size, and for most cases, an increase of salt concentration causes a decrease of crystallite size.

Rodriguez and Fernandez-Garcia [24] reported crystallite sizes considerably smaller in the range of 7–9 nm for copper (II) oxide. The crystallite size of the CuO nanoparticles depends on the preparation method used. According to Aparna et al. [25], the average particle diameter is: (i) about 10 nm when prepared by sonochemical; (ii) 1–10 nm by the sol-gel method; (iii) 3–9 nm in the decomposition of copper acetate; (iv) 15–20 nm in solid-state reactions; and (v) 4 nm when using the electrochemical method.

It is observed that any material which contains particles or agglomerates of smaller sizes than 100 nm, or films, and filaments of this range are considered nanostructured materials [26]. For Rodriguez and Fernandez-Garcia [24] nanoparticle sizes definition includes the range from 1 to 1000 nm, which can be amorphous, fully crystalline or small aggregates of crys-
Fig. 4 – Nanoparticles obtained with CuSO₄·5H₂O, concentration 0.2 M and temperature 50 °C: a) and b) bright field images; c) detail of the formed particle; d) dark field image.

3.4. Transmission electron microscopy (TEM), nanoparticles prepared with copper sulfate

In Figs. 3 and 4, TEM microphotographs for nanoparticles obtained from CuSO₄·5H₂O precursor are presented. The nanostructured particles synthesized at 50 °C at both concentrations have shown the same morphology. Agglomerates were built with an average crystallite size between 10 and 11 nm. The rod-like crystals formed agglomerates with 190 nm width in the center and thinner on the ends. Larger star-like agglomerates formed three-dimensional clusters with diameters ranging from 400 to 700 nm. In Fig. 3c, the image was carried out by electron diffraction and indicated a nanostructured and polycrystalline material.

With temperature increasing to 75 °C (Figs. 5 and 6), larger crystallites with an average size of 14.5 nm were formed (0.2 M precursor concentration). The sample prepared with 0.1 M precursor (Fig. 5) showed irregular structure, probably caused by the presence of those chemical species: brochantite (Cu₄(SO₄)₂(OH)₂) and posnjakite (Cu₁₂(SO₄)₆(OH)₆H₂O), detected in XRD analysis.

The rods formed in sample D (Fig. 6) had an average width of 220 nm and an average length of 800 nm, forming larger structures than the synthesis carried out at 50 °C.

The reaction temperature also has influenced on the preferred orientation of crystal growth. At 50 °C, the crystallites agglomerated and built a three-dimensional structure with 6 edges, and at 75 °C, the crystal growth followed a linear direction, forming small particles similar to a rod.

3.5. Transmission electron microscopy (TEM), nanoparticles prepared with copper chloride

Sample E was selected for TEM analysis and results obtained with the precursor CuCl₂·2H₂O, are shown in Fig. 7. Agglomerates were visualized in this sample also, and crystallite size was greater than those obtained with copper sulfate as a precursor (considering the same conditions: 50 °C and 0.1 M). In this case, the crystallites obtained had an average size of 21 nm and formed rod-like structures with an average size of 220 nm width and 830 nm in length, with subsequent cluster formation in three-dimensional structures.

The crystallite size calculated from TEM micrographs and the values obtained by the Scherrer method revealed good correlation as presented in Table 3. The major differences observed in samples C (Fig. 5) and E (Fig. 7) represents synthesis conditions where the nanoparticles were a mixture of copper oxide and different contaminants.
Fig. 5 – Nanoparticles obtained with CuSO\(_4\)·5H\(_2\)O, concentration 0.1 M and temperature 75 °C: a) and b) bright field images of formed particles; c) and d) bright and dark field images of a nanostructured particle.

Fig. 6 – Nanoparticles obtained with CuSO\(_4\)·5H\(_2\)O, concentration 0.2 M and temperature 75 °C: a) bright field image; b) and d) bright and dark field images of a nanostructured particle prepared; c) crystallite details.
3.6. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX)

3.6.1. Copper sulfate as a precursor
Samples A, B, C and D (CuSO₄ precursor) are presented in Figs. 8, 9, 10, and 11, respectively. The results of analysis by energy dispersive spectroscopy indicate the presence of only copper and oxygen in the samples developed with this precursor. The other two peaks appearing in Figs. 8 (b), 9 (b), 10 (b) and 11 (b) correspond to the presence of carbon and gold used, respectively, in the carrier tape and covering the samples.

3.6.2. Copper chloride as a precursor
Sample E (Fig. 12a) prepared with precursor copper chloride (CuCl₂) was selected for this analysis (synthesis conditions: 50 °C and 0.1 M). In EDX results, Fig. 12b, a presence of the atom Chlorine is confirmed. This is due to the formation of clinoatacamite and its polymorphs in the sample, as confirmed by XRD results. The same peaks of carbon and gold are also observed because of experimental conditions.

3.7. Thermal analysis (TGA and DSC)
Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) was performed on samples synthesized at 50 °C and with 0.2 M of precursor salt concentration (Samples B and F) and the results are exhibited, respectively, in Fig. 13a and b.

According to Patnaik [27], the heating when the temperature is above 800 °C converts the copper (II) oxide into copper (I) oxide. Above this temperature, copper (I) oxide becomes the more stable phase. For this reason, the samples were tested until 1000 °C to observe the thermal stability of the obtained CuO samples.

Sample B (Fig. 13a) produced with the precursor copper sulfate, showed a weight loss of about 3.4 %; in the temperature range between 25 and 136 °C, probably due to the vaporization of water contained in the sample. There is a small signal at 283 °C associated with a weight loss of 1.15 % due to crystallization water removal; these events are summarized in Table 4 (peaks values estimated in OriginPro 8).

Sample B exhibited very good thermal stability, losing only 5.6 % of its mass until the decomposition of CuO started at about 900 °C. The difference in CuO decomposition temperature, when compared with the literature, was probably an effect of the resolution used (10 °C / min).

<table>
<thead>
<tr>
<th>Precursor</th>
<th>Sample</th>
<th>Scherrer (nm)</th>
<th>TEM (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuSO₄·5H₂O</td>
<td>A</td>
<td>10.7</td>
<td>10.7</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>10.0</td>
<td>10.9</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>13.8</td>
<td>36.9</td>
</tr>
<tr>
<td>CuCl₂·2H₂O</td>
<td>D</td>
<td>14.1</td>
<td>14.6</td>
</tr>
<tr>
<td></td>
<td>E</td>
<td>14.5</td>
<td>20.8</td>
</tr>
</tbody>
</table>
Fig. 8 – CuO nanoparticles obtained with the precursor CuSO₄, conc. 0.1 M and temperature 50 °C: a) micrograph; b) elemental analysis by EDX.

Fig. 9 – CuO nanoparticles obtained with the precursor CuSO₄, conc. 0.2 M and temperature 50 °C: a) micrograph; b) elemental analysis by EDX.

Fig. 10 – CuO nanoparticles obtained with the precursor CuSO₄, conc. 0.1 M and temperature 75 °C: a) micrograph; b) elemental analysis by EDX.

Fig. 11 – CuO nanoparticles obtained with the precursor CuSO₄, conc. 0.2 M and temperature 75 °C: a) micrograph; b) elemental analysis by EDX.
**4. Conclusion**

Nanoparticles of copper oxide nanoparticles (II) were made from two precursors: copper sulfate (CuSO₄•5H₂O) and copper chloride (CuCl₂•2H₂O) using concentrations of 0.1 M and 0.2 M; and temperatures of 50 °C and 75 °C.

The characterization by X-ray diffraction (XRD) showed the presence of a crystalline phase of copper (II) oxide in the samples, using copper sulfate as a precursor. However, with the same precursor, increasing the temperature to 75 °C caused the appearance of compounds like brochantite (Cu₄(SO₄)(OH)₆) and Posnjakite (Cu₄(SO₄)(OH)₆•H₂O) together with copper (II) oxide phase, only with precursor salt concentration of 0.1 M.

All samples prepared with the copper chloride precursor has been identified as a mixture of crystalline phases, with the presence of chlorinated compounds with chemical formula Cu₂(OH)₃Cl, and a copper (II) oxide phase. These chlorinated chemical compounds exhibited a polymorphism phenomenon and its most stable form is called clinoatacamite.

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**Fig. 12** - CuO nanoparticles obtained with the precursor CuCl₂, conc. 0.1 M and temperature 50 °C: a) micrograph; b) elemental analysis by EDX.

**Fig. 13** - Thermal analysis (TGA and DSC) of CuO nanostructured particles: a) sample B, precursor CuSO₄; b) sample F, precursor CuCl₂. Synthesis conditions: temperature 50 °C and precursor salt concentration 0.2 M.

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**Table 4** - Results obtained experimentally in the thermal analysis of sample B: precursor CuSO₄. Synthesis conditions: temperature 50 °C and precursor salt concentration 0.2 M.

<table>
<thead>
<tr>
<th>Event</th>
<th>Temp. Range (°C)</th>
<th>Central Peak (°C)</th>
<th>Weight loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.6–136.4</td>
<td>58.2</td>
<td>3.38</td>
</tr>
<tr>
<td>2</td>
<td>230.8–349.0</td>
<td>283.1</td>
<td>1.15</td>
</tr>
<tr>
<td>3</td>
<td>909.2–995.6</td>
<td>943.8</td>
<td>5.98</td>
</tr>
</tbody>
</table>

The thermal behavior of sample F (Fig. 13b) which is a mixture of copper (II) oxide and chlorinated compounds of chemical formula Cu₂(OH)₃Cl, showed less thermal stability and larger weight loss in the temperature range observed. The different weight loss results observed in Figs. 13a and b are probably due to chlorine gas, eliminated by heat in sample F.

Performing the analysis by the DSC curve, three endothermic peaks were observed until 250 °C, probably induced by dehydration of the nanostructured crystals. The five events exhibited by this sample are displayed in Table 5 (data estimated in OriginPro 8). Another endothermic peak appeared at about 800 °C, associated with a small weight loss (0.36 %), indicating a probable crystalline transition, followed by a massive loss indicating material decomposition.

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**Table 5** - Results obtained experimentally in the thermal analysis of sample F: precursor CuCl₂. Synthesis conditions: temperature 50 °C and precursor salt concentration 0.2 M.

<table>
<thead>
<tr>
<th>Event</th>
<th>Temp. Range (°C)</th>
<th>Central Peak (°C)</th>
<th>Weight loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.9–107.3</td>
<td>66.1</td>
<td>15.84</td>
</tr>
<tr>
<td>2</td>
<td>141.4–154.1</td>
<td>147.8</td>
<td>0.63</td>
</tr>
<tr>
<td>3</td>
<td>201.3–252.0</td>
<td>218.5</td>
<td>3.25</td>
</tr>
<tr>
<td>4</td>
<td>782.7–800.1</td>
<td>796.1</td>
<td>0.36</td>
</tr>
<tr>
<td>5</td>
<td>943.7–985.7</td>
<td>970.8</td>
<td>11.20</td>
</tr>
</tbody>
</table>
The crystallite size was calculated by Scherrer formula and lower average crystallite sizes were obtained using the copper sulfate solution as a precursor. It was observed that the increase of reaction temperature promotes the development of the chlorinated crystalline phases during the nanostructured material formation.

Thermal analysis revealed the different behavior of the nanostructured materials obtained. When prepared with copper sulfate salt the material exhibited only 5.6 % weight loss until decomposition temperature of CuO. As a comparison, the sample made from copper chloride has lost more than 30 % of the mass, considering 800 °C as the decomposition temperature, probably due to chlorine gas elimination.

The analysis of its elemental composition by EDX evidenced the presence of chlorine, copper, and oxygen in the samples prepared with the precursor copper chloride, and, only copper and oxygen in samples prepared from copper sulfate.

The morphology of the nanoparticles and their dimensions were observed in TEM photomicrographs, in which the nanoparticles were presented as aggregates of crystallites in the form of a small rod, organized into three-dimensional structures with average dimensions differing according to synthesis conditions. The samples synthesized at 50 °C precursor with the copper sulfate had structures with smaller dimensions than using 75 °C as reaction temperature, thereby forming a more interesting material for future applications.

With the use of copper chloride as a precursor, leaf-like nanoparticles were prepared, which then formed larger three-dimensional structures with about 830 nm in length, similar to those found with the other precursor.

The average crystallite size showed a good correlation between the values obtained by the Scherrer method and TEM photomicrographs. The formation of a single-phase copper oxide (II) in the synthesized material from the precursor copper sulfate, using appropriate reaction conditions allows the preparation of copper oxide nanocrystals for numerous applications.

The coprecipitation method used revealed to be fast and simple, using only common materials of a chemical laboratory and with low reaction temperatures.

**Conflicts of 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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