

Research Paper

The Simulation on the Phase Formation of Bi-Ba-Ca-Cu-O high Temperature Superconductor

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ABSTRACT

The high-temperature superconductor like $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ was prepared using the solid-state reaction method at different calcination temperatures including 780, 800, and 850 °C. X-ray diffraction technique was used to define crystal structure formation as a function of calcination temperature. It was found a tetragonal phase was obtained with lattice constants of $a=b=4.2967$, and $c=36.5248$ Å. It was found that there is a limited difference in the scattered peak intensities making a partial variation in the fraction volume of the phase formation. Such that the volume fraction of a high phase superconductor recorded 73.76% for the Bi-2223 phase at calcination temperature of 800 °C. There is high intensity for the most probable peaks in the prepared sample at a temperature of 800 °C mentioned by peaks (010), (110), and (0116) related to the tetragonal phase. Their intensities were twice incomparable with the peaks that appeared at calcination temperature at around 850 °C. That is returned to high tetragonally and high stability in the atomic distribution within the unit cell. The structure simulation was applied due to the experimental X-ray patterns to investigate the formation of tetragonal shape via a unit cell. A Williamson-Hall method was used to show the crystallite size and the possible strain in the unit cell. It was found the lowest value of strain about (0.0005) appeared at calcination temperature of 800 °C. SEM and EDS had accomplished the surface morphology and elements concentration, their results were confirmed with XRD analysis.

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1. Introduction

Two familiar families of superconductor copper oxides were studied in different global laboratories specialized in high-Tc superconductivity. The first type was related to $\text{La}_{2-x}\text{A}_x\text{Cu}_{4-y}$ (A=Ca, Sr, Ba) prepared by solid-state reaction and studied firstly by Bednorz and Muller [1] and their derivative like $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ [2]. The second family was condensed on the $\text{Bi}_2\text{Sr}_2\text{Cu}_2\text{O}_{7+\delta}$ compound mentioned by Michel et al. [3]. The last one belongs to the Bi-Sr-Ca-Cu-O system studied by Cheetham et al. [4]. These compounds showed a multi perovskite structure ordering by oxygen-deficient through multilayers of CuO_5 and CuO_4 polyhedral via Bi-planes. They have exhibited multi phases mentioned by low phases like (2201), (2212), and high phases remarked by (2223); each phase has different diffracted peaks. The effect of multiphase is clear in the critical temperature and the conductivity mechanism [5]. X-ray diffraction pattern showed a pure phase of $\text{Bi}_2\text{Sr}_2\text{Cu}_2\text{O}_{7+\delta}$ as a multi perovskite structure with orthorhombic unit cell and lattice parameters of $a=5.32$, $b=26.6$, $c=48.8$ Å [3]. On the other hand, the system of Bi-Al-Ca-Sr-Cu-O recorded superconductivity up to 114 K [6], it showed a multiphase of multi perovskite unit cells. They observed unusually high-Tc superconductor oxides; it is able for different applications and might be higher and stable Tc-values soon. Previously many papers discussed the formation of a high-temperature superconductor [7,8]. They were discussed the required phase experimentally depending on many parameters applied. The thing is different in this research concentrating on the simulated results concluded by the experimental data. So, it is considering the base on conclusion the phase required of high-temperature superconductor.

It is well known that the Bi-Sr-Ca-Cu-O family contains three phases, starting from low phase (2201), (2212), and (2223) high phase; these are different in the diffraction process. The phase formula of the $\text{Bi}_2\text{Sr}_2\text{CuO}_6$ compound is (2201) without Ca-ion concentration; it was exhibited a critical temperature of about 20 K. The structural formula of the second phase is $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ compound with a critical temperature of 90 K. The formula $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ compound represents the third phase; it has a higher critical temperature than the previous two phases of about 110 K [9]. The possibility of partial substitution by isovalent ions in the main composition $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ was discussed previously [10-13] to improve the superconducting properties. The main feature that has distinguished the creation of high phase was related to the volume fraction [14]; they have discussed the volume fraction ratio created and their effect on the critical temperature. The phase

formation depends on the calcination process represented by the calcination temperature and duration [15]. They found the calcination temperature was about 820 °C through the 24 h duration. So that the challenge is reducing the calcination temperature and its duration to form a high phase superconducting compound with a high volume fraction.

The new thing was discussed in this research represented by completely inserting Ba-ions instead of Sr-ions. Both ions (Sr, Ba) have the same valency, and both are in the same column in the periodic table. The difference between them is related to the atomic radius; it is 253 pm for Ba atom whereas 219 pm for Sr-atom. That was affected by the dimension of a unit cell. This study was condensed on a simulation in the structure of the composition $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ prepared by solid-state reaction under different calcination temperatures and the possible change on the unit cell dimension and atomic positions. This research provides more information on the phase formation required to understand the mechanism of conductivity later. A solid-state reaction is divided into calcination and the sintering process. The calcination process is the phase formation process, so it is necessary to understand the mechanism of phase formation under different calcination temperatures via suitable software required for simulation. Williamson-Hall method is a method used to calculate the crystallite size and elastic strain in a polycrystalline structure depending on the following equation [16].

$$\beta \cos\Theta = k \lambda/D + [4\epsilon \sin\Theta] \quad (1)$$

The relation between ($\beta \cos\Theta$) on the y-axis and ($4 \sin\Theta$) on the x-axis showed a straight line, such that the interception with y-axis means the crystallite size (D) and the slope of the straight line exhibited the elastic strain (ϵ) might happen in the unit cell. This function is depending on the peak width at certain diffracted angles (2θ), which is relating to crystallite size and the strain that might be happened in the unit cell. The size-strain plot (SSP) is a combination of the Lorentzian function and Gaussian function from XRD peak chart. Where the crystallite size was labeled by Lorentz function and strain related to Gaussian. The lattice strain was attributed to expansion or shrinkage in the lattice constants, which is resulting from the stress coming through X-ray radiation applied.

2. Experimental procedure

The samples of the superconducting compound $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ were prepared by the solid-state reaction method. The principal materials were used must be high purity and imported from the Merck

industry; they were $(\text{BiO})_2\text{CO}_3$, BaCO_3 , CaCO_3 , and CuO . It used suitable weight for the above compounds by the ratio 2:2:2:3, and mixed many times to make the homogeneity. The mixture was crushed using an agate slurry with the addition of some drops of propanol-2. The powder was calcined at different temperatures including 850, 800, and 780 °C for 24 h in the air. The aim of calcination is the creation of a required superconducting phase. So, it is necessary to understand which calcination temperature is suitable to show the presence of Bi-2223 high superconducting phase. For this purpose, X-ray diffraction at room temperature was used to define the accurate difference in a structural phase obtained. The X-ray diffractometer type Phillips with Cu-K α source at the wavelength of 1.5405 Å. Structural behavior of the compound $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ at different calcination temperatures was analyzed by using suitable software to show the limited variation that appeared in the perovskite unit cell during the lattice parameters (a, b, and c) and the position of atoms. The lattice constants and atomic positions are related to Miller indices mentioned by the diffracted peaks intensities at certain diffracted angles. So the aim of this research is dealing with a favor calcination temperature is applying to produce a high phase superconducting state labeled by Bi-2223. The SEM and EDS type Inspect S50 are suitable to show the surface morphology and the contents of the element in the mixture.

3. Results and Discussion

After the successful preparation of the sample under study, X-ray diffraction is necessary to show the required phase incomparable with a database of $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$. The high-temperature superconducting samples were prepared at different calcination temperatures including 850, 800, and 780 °C. This research aims to exhibit which calcination

temperature is favorable to get the required phase of $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ high-temperature superconductor compound. The data showed a pure tetragonal phase formed by $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ at a calcination temperature of 800 °C. The X-ray diffraction simulation during CrystDiff-software produced lattice constants (a, b, and c). Endevar-software had conducted the simulation on the last data to conclude all information related to the atomic position in the unit cell shape. The samples were prepared at calcination temperature of 780, and 850 °C showed a similar pattern to that at calcination temperature of 800 °C, but there is a limited variation in the intensity of the peaks. It is well known that the intensity of the peaks relates to the concentration of crystallites in the structure. Typically, most peaks have the same concentration except the peak (0012) in a tetragonal phase. That means the high concentration appeared at 800 °C temperature

The results were showed a tetragonal phase with lattice constants of $a=b=4.2967$, and $c=36.5248$ Å, the volume of a unit cell is 674.07 Å³, and space group P4/mmm. The calculated density is 2.767 g/cm³, and the compound's molecular weight is 1123.4. These results contradict Shmary et al. [17], who made a refinement on the XRD-pattern of $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ and concluded a lattice constant of $a=5.4113$, $b=5.4091$, and $c=37.0821$ Å with orthorhombic phase. The difference point concentrated on the group symmetry depends on the refinement. Secondly, it might be related to the total replacement of Ba-atom instead of Sr-atom. The distinguishable points in our results, it depends on the structure simulation by two software depends that make our results are more accurate with respect to Wadi et al. [18], they were determined the lattice constants depends on the comparison with ASTM-data only.

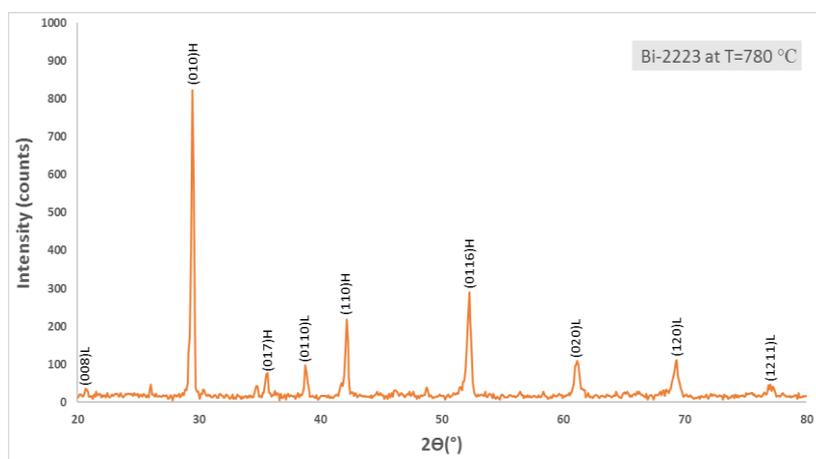


Fig. 1. Indicate X-ray diffraction pattern for Bi-2223 at calcination temperature of 780 °C

The difference in the calcination temperature (780, 800, and 850 °C) was appeared in the peak intensities, as shown in Figs 1-3, respectively. It is well defined that most peaks are symmetrical, but there is a difference in their intensities. In addition, the peaks are classified into two types mentioned by

H related to high phase and L function to low phase. This classification is a function of the value of T_c if it is high or low. It was exhibited that the highest diffracted intensity detected at calcination temperature of 800 °C, as shown in Fig. 2.

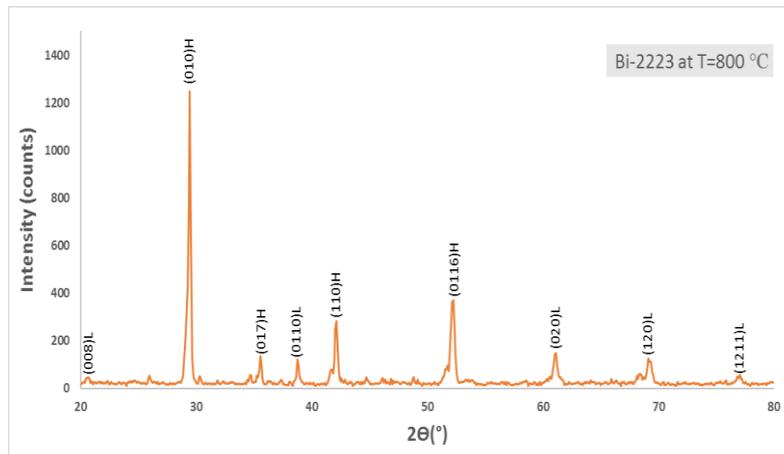


Fig. 2. Indicate X-ray diffraction pattern for Bi-2223 at calcination temperature of 800 °C

The X-ray diffraction patterns for the sample prepared at 850 °C was showed low intensities in some peaks, and others disappeared, as shown in Fig. 3. The most probable peaks at a high intensity are mentioned by (010), (110), and (0116) for the sample calcined at 800 °C related to the tetragonal phase.

There is a substructure predicated within the tetragonal unit cell is represented by two tetrahedron structures forming Bi-O₅ and the connection layer represented by Cu₂-O as appeared in a simulated unit cell (Fig. 4).

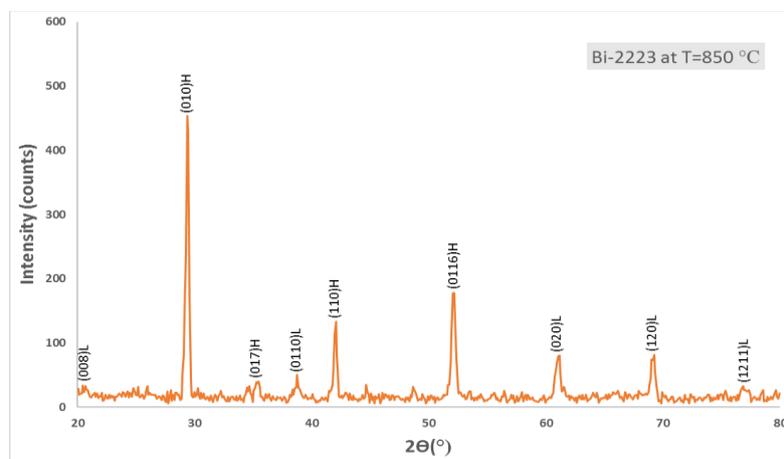


Fig.3. Indicate X-ray diffraction pattern for Bi-2223 at calcination temperature of 850 °C

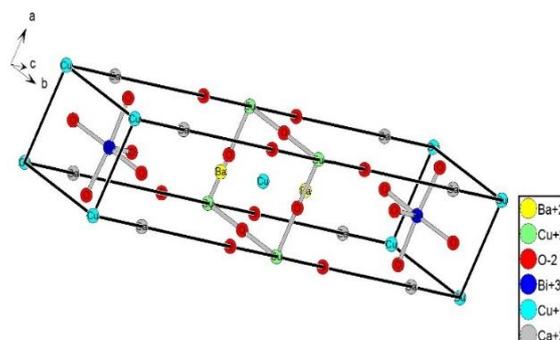


Fig. 4. Explain the simulated unit cell of superconductor compound

The simulated unit cell is the primary purpose of defining the position of atoms producing the tetragonal shape, as presented in Table 1. It is essential to know more information about the structural phase, as the bonds between atoms, because it is the origin of the energy stored in the structure, and the last is responsible for the conductivity mechanism in superconductor compounds [19]. The general information was predicated from the simulated unit cell was the presence of tetrahedron structure produces a net of polarization on both sides. It is an intense

polarization producing a built-in electric field, which is a reason to accelerate the charge carrier created from the Cu₂-O layer through c-axes. On the other hand, the electric field created by the tetrahedron enforced the coupling between sequences of Cu₂-O layers. The second thing, the above Miller indices play an essential role in transporting charge carriers through the unit cell. They will act as interacting layers between tetrahedrons substructure through the Cu₂-O layer. The distribution of atoms through the tetragonal unit cell was predicated in a simulated unit cell.

Table 1. Indicate the positions of atoms in a simulated unit cell of Bi₂Ba₂Ca₂Cu₃O_{10+δ} compound

atoms	X	Y	Z
Bi1	0.5	0.5	0.0806
Ba1	0.5	0.5	0.3848
Ca1	0	0	0.1329
Cu1	0	0	0.5
Cu2	0.5	0.5	0.5
Cu3	0	0	0
O1	0.5	0.5	0.1419
O2	0	0	0.3738
O3	0	0.5	0.5
O4	0	0.5	0.0754

It is well known that the calcination temperature is critical to form the required phase for a high-temperature superconductor. Then the volume fraction is a good sign to show the presence of the (2223) high phase. For this reason, it is necessary to apply the following equation [20].

$$Bi[22(n-1)n]\% = \frac{\sum I_{Bi-[22(n-1)n]}}{\sum I_{Bi-2223} + \sum I_{Bi-2212} + \sum I_{Bi-2201}} \times 100 \quad (2)$$

where [n=1,2,3] indicates the phases 2201, 2212 and 2223. It is clear that the volume fraction ratio

is a function of calcination temperature and its duration, as presented in Table 2. The results were showed the highest volume fraction recorded at calcination temperature of 800 °C with a limited ratio of volume fraction related to low phase (2201), and (2212). This value is favor than that was recorded by Garnier et al. [15]. They observed that a calcination temperature of 820 °C was suitable for obtaining a volume fraction of 79% of Bi-2223 high phase for 24 h duration. So, the difference in the calcination temperature is a benefit for saving energy.

Table 2. Indicate the results of Williamson-Hall calculation and volume fraction ratio

Calcination temperature (°C)	Crystallite size nm	Structure strain	Phase volume fraction		
			2201 phase	2212 phase	2223 phase
780	92.43	0.0025	8.206	20.959	70.834
800	34.6613	0.0005	7.248	18.982	73.768
850	19.8064	0.0009	11.274	23.148	65.576

It is necessary to understand the mechanism of crystallites creation, which is the seed to form the required phase. The calculation of crystallite size and elastic strain on the proposed structure had been done using Williamson-Hall measurements,

as shown in Figs 5-7. There is a change in crystallite size as a function of calcination temperature, as shown in Table 2. The X-ray patterns showed the presence of polycrystalline that had much orientation mentioned by multiple

diffracted peaks. These orientations are represented by different grains, including. The components of each grain include many crystallites sizes; those were discussed by Williamson-Hall calculation. So that each peak is a function of a certain crystallite size different

from others, which means the low intensity relates to the low density of those orientations represented inside the grains. It is well known that crystallite size is a part of grain size. On the other hand, each grain contains many crystallites, and many grains produce particle size.

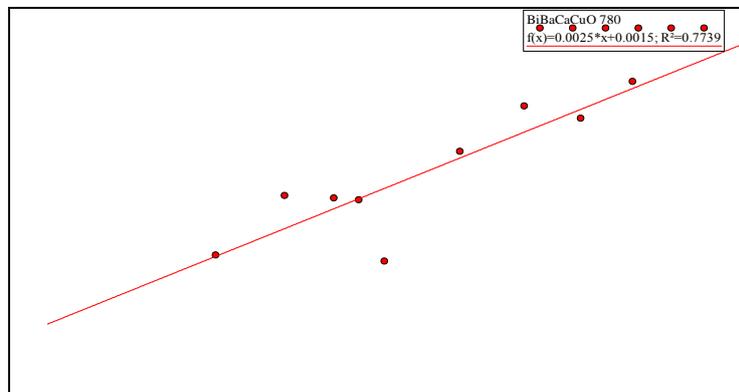


Fig. 5. Indicate the Williamson-Hall calculation for sample calcined at 780 °C

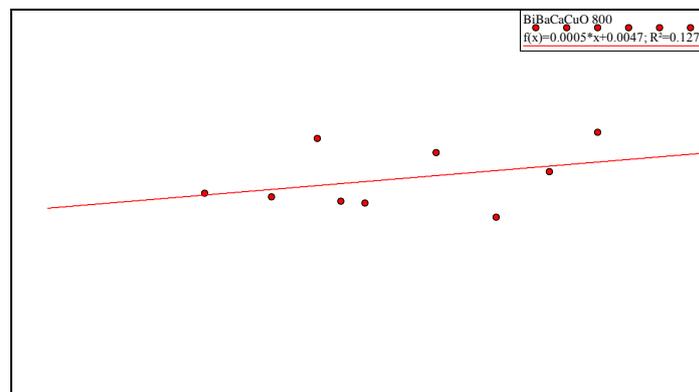


Fig. 6. Indicate the Williamson-Hall calculation for sample calcined at 800 °C

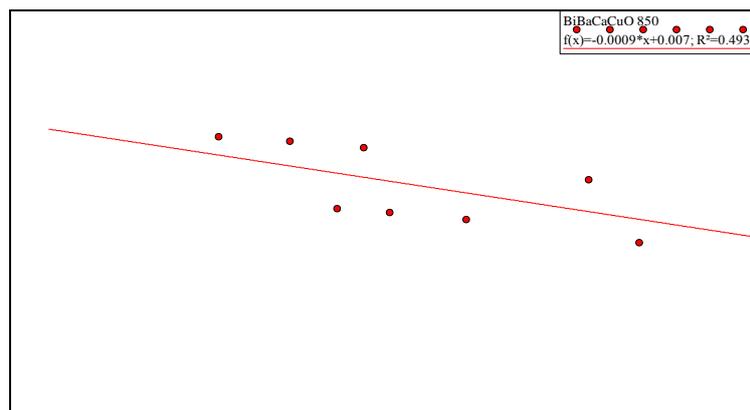


Fig. 7. Indicate the Williamson-Hall calculation for sample calcined at 850 °C

Table 2 explains the reduction of crystallite size as a function of calcination temperature. This reduction is related to the decrease in the grain size and possible decreasing in the grain boundaries, which were considered a positive effect in the scattering mechanism and discussed by SEM and EDS analysis later. On the other hand, reducing grain boundaries tends to make a high dense sample after sintering.

The benefit of things presented in Table 2 is the strain in the structure. It is well defined that is elastic strain happens due to external effects. So the possible strain in the lattice constants appeared as a function of calcination temperature; it is clear that a low strain is related to the high stability of the phase obtained at T=800 °C. That confirms the favor calcination temperature to produce the pure tetragonal phase due

to the lowest value of strain about 0.0005, which is an approach to a value concluded by Al-Khafaji *et al.* [21]. Whereas, there is a lower crystallite size value of about 34.6613 nm, which is a return to half-width of mentioned peaks if it is wide or narrow peaks in

addition to their intensity. It is comparable with previous results recorded by Al-Khafaji *et al.* [21]. They recorded the crystallite size by Williamson Hall of about 56.395 nm for $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ at $x=0$.

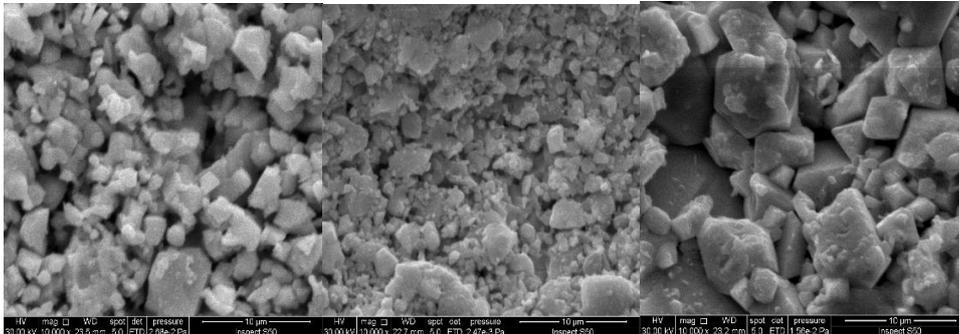


Fig. 8. SEM photo magnified at 10 μm , for the sample $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ calcined at different temperature; (a) 780 $^\circ\text{C}$, (b) 800 $^\circ\text{C}$, (c) 850 $^\circ\text{C}$

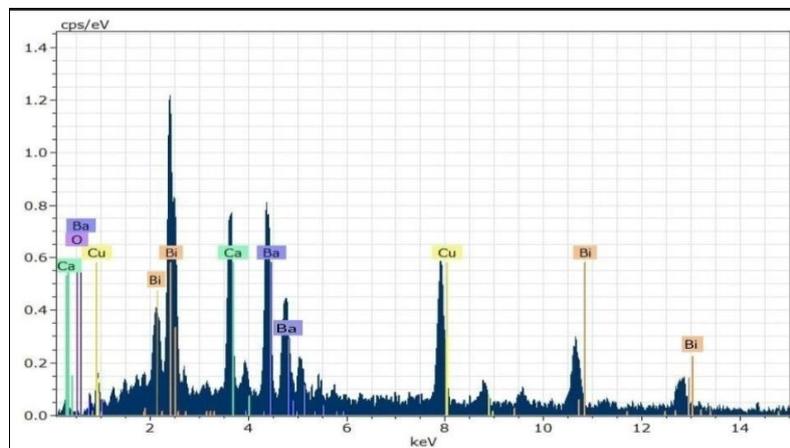


Fig. 9. Indicate EDS-pattern for Bi-2223 at calcination temperature of 800 $^\circ\text{C}$

The SEM image exhibits the surface morphology at different calcination temperatures, as shown in Fig. 8. Obviously, SEM pictures showed high homogeneity and appearance of dark and lighted regions. The dark regions were dominant to heavy elements content like Bi-ions and CuO layer including. This is proof of increasing O-ions concentration in the compound. The decreasing of the dark region is related to decreasing of diffracted intensity peaks, as discussed in XRD-analysis. Then it is a confirmation that the enhancement of a regular crystalline structure of high phase (2223) signed by tetragonal phase, as discussed before. The coincidence in results with Gitan *et al.* [13], will emphasize the formation of the high phase mentioned by the Bi-2223 high-temperature superconductor. Fig. 8b showed the lowest concentration of the dark region; on the other hand, the tetragonal phase enhancement in Fig. 8c, during a crystalline shape in 3D. That means increasing calcination temperature of 850 $^\circ\text{C}$ is powerful in producing alike single crystal. The last result is confirmed with a high volume fraction ratio of about 66%. The above results are in

agreement with the EDS-analysis for the sample was prepared at calcination temperature of 800 $^\circ\text{C}$, as shown in Fig. 9. The main elements in the structure are clear, as mentioned in the experimental part. It is confirmed with the simulated tetragonal shape and the distribution of those atoms in the simulated shape. The correlation between all analysis, calculation, and simulation has exhibited the preparation condition's success in the formation of the Bi-2223 high superconductor phase. There is no remarkable change in the EDS chart for the samples were prepared at calcination temperature of 780, and 850 $^\circ\text{C}$; they showed the same ratios of all elements.

4. Conclusion

The obtainable results were proved the favor calcination temperature was 800 $^\circ\text{C}$ to form a tetragonal phase of $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$. It is suitable to prepare high-temperature superconductors type Bi-2223 after the calcination process. The results of simulation on X-ray diffracted patterns give more details on the structure type and the tetrahedral bonds, which are effective on the conductivity

mechanism through c-axes within the unit cell. The Williamson-Hall calculation gives us more information about the stability of which sample is fixed through the lowest value of elastic strain, the dimension of crystal structure. The high tetragonal phase appeared for the sample prepared at calcination temperature of 800 °C. The second thing, this simulation on the X-ray patterns and Williamson-hall method, is considered a good tool to show the stability of the phase prepared under certain conditions. The SEM and EDS analysis were a good agreement with the above results to confirm the phase formation of Bi-2223.

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