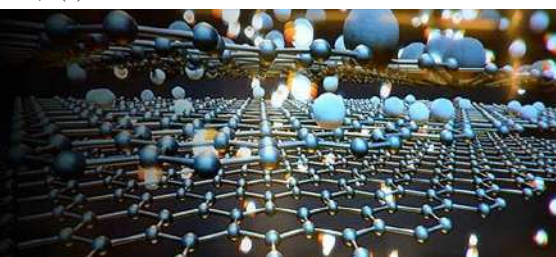


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Facile synthesis process & FTIR characterization study of prepared silicate powder sample

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Abstract

Due to a rapidly growing market and lighting industries for their huge variety applications in traffic signs, decoration, and textile printing, among others, alkaline earth such as Calcium (Ca), Strontium (Sr), and Barium (Ba) rare-earth doped silicates have also been studied as persistent luminescence-phosphors. When compared to a sulphide materials previously utilised, silicate phosphors achieve superior results, including longer and brighter light emission, quicker preparation, and reduced cost. Luminescent materials, which are essentially phosphors, and other ceramic products such nano materials are already of more specifically due to the combustion process. This is mostly due to the fact that at relatively low temperatures and for relatively brief times, tiny particle morphologies, multi-component, crystalline, and homogeneous products can be produced. Such a procedure often involves a redox reaction between an organic fuel and an oxidant, such as metal nitrates. A $\text{Ba}_2\text{MgSi}_2\text{O}_7$ powder sample was successfully made using the low temperature classical combustion synthesis method. Through FTIR, the real formation of this phosphor was discovered. An FTIR spectrum was recorded with the help of Bruker Alpha Fourier Transform Spectroscopy. For investigating the functional groups (4000 to 1400 cm^{-1}) as well as the finger print area (1400 to 400 cm^{-1}) of synthesized phosphor through mixing the sample with analytical grade potassium bromide (KBr) with pallet preparation. The present investigation till be made with respect to the Preparation of Barium magnesium silicate phosphor and Measurement of optical properties such as Fourier Transform Infra-red Spectroscopy (FTIR) of prepared phosphors.

Keywords: $\text{Ba}_2\text{MgSi}_2\text{O}_7$, Fourier Transform Infra-red Spectroscopy (FTIR)

1. Introduction

Mellites are a large crystal-structured group of compounds whose represented by means of usual structure representation [i.e. $\text{M}_2\text{A}^1\text{B}^2\text{O}_7$, where M= Ba (Barium), Sr (Strontium), Ca (Calcium); A^1 = Mg (Magnesium), Zn (Zinc), Cu (Copper), Mn (Manganese), Co (Cobalt); B^2 = Ge (Germanium), Si (Silicon)]. White light emitting diodes (WLEDs) are recognized as the new-generation lighting source because they are high-light extraction efficiency and brightness, long-lifetime, and environmental friendliness, cheaper etc. An effective host material that reduces the likelihood of the killer centre trapping excitation energy is $\text{Ba}_2\text{MgSi}_2\text{O}_7$ phosphor. The FTIR spectra have been used to evaluate the silicate (SiO_4) absorption bands and various functional groups of related pure compounds in order to well agree for actual clarifications.

Due to benefits including superior thermal strong light emission and chemical stability, rare earth doped silicate materials have recently undergone extensive research into their luminous characteristics. These phosphors might develop into promising white light emitting diode (WLED) phosphor choices. Phosphor converted white light emitting diodes are among these applications' top options for solid state lighting due to their exceptional qualities, including long operational lifetimes, energy efficiency, high brightness, and compactness and environmentally behaviour.

The key factor driving an increase in phosphor research activities in the twenty-first century was their excellent application orientation. Two orders are typically where the majority of phosphor research is focused. The first step is to identify the trapping-de-trapping process responsible for the persistent luminescence. The creation and development of new long afterglow phosphors that offer a wide spectral area from visible to IR wavelengths constitutes the second strategy. The silicate-based phosphors possess a variety of properties, including good water-repellence and physical and chemical stability. It also has a broad range of commercial applications and offers a number of advantages, including brightness,

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heat resistance, abundance, affordability, and a selection of luminescent colours from blue to red. Material scientists are very interested in the silicate host due to the widespread use of phosphors with lengthy afterglows. The $\text{Ba}_2\text{MgSi}_2\text{O}_7$ phosphor in this paper was made using a low temperature combustion synthesis process. Fourier Transform Infra-Red Spectroscopy (FTIR), which was also described, was used to analyse the material optically.

2. Combustion Synthesis Technique

For the fabrication of the powder sample in our current experiment, we used the low temperature combustion synthesis technique. The phosphors are the more well-known name for the luminous substances. Phosphor is typically a certain kind of thing. This causes the illumination event to be seen. The advantages of the combustion synthesis method are described, along with a brief explanation of its synthesis principles and specific procedures. As an important synthetic method for the synthesis and processing of promoted ceramics (in both anatomical and functional forms), activators, farraginous, alloys, intermetallic, and nano products, combustion

synthesis has recently gained prominence. By happily disposing of a significant volume of single phase and well crystallised multi factor oxides, urea and glycerine, two well-known fuels, have demonstrated the adaptability of this approach. An exothermic reaction that causes a rise in temperature is also a part of this process. A self-propagating low temperature synthesis method is sometimes used to describe this process because once the pioneers are burning; the energy needed for the combustion reaction is recovered by the reaction itself. Combustion-produced phosphors have less dense and fluffy structural characteristics. The process also produces extra fine particles, which are combined with growing gas levels. Agglomerates are not broken down or reduced to fine particles as the maximum amounts of gases are released. This method outperformed other conventional ceramic processing techniques in a wide range of other ways, including: (i) Low reaction time; (ii) Inexpensive processing tool; (iii) Harnessing the chemical energy of the reactants to make the final material in a single step (iv) Absence of volatile substances and high quality components.

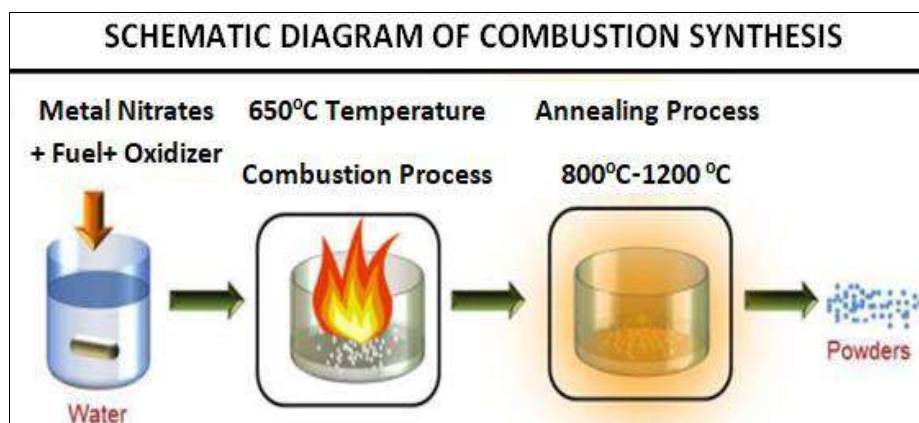


Fig 1: Flow Chart of Combustion Synthesis Process.

3. Experimental Procedure

3.1 Sample Preparation

We applied in our experiment (Fig: 3); $\text{Ba}_2\text{MgSi}_2\text{O}_7$ sample were successfully synthesized via combustion synthesis technique. All initial reagents have taken with (99.99%) purity, $\text{Ba}(\text{NO}_3)_2$ (AR), $\text{Mg}(\text{NO}_3)_2$ (AR), $\text{SiO}_2 \cdot \text{H}_2\text{O}$ (AR). Very little quantities were used urea (NH_2CONH_2) as a combustion fuel and H_3BO_3 (boric acid) (AR) as a flux. The weighted ingredients were combined to create a thick paste in an agate mortar and pestle. After being deposited into the cylindrical silica crucible, the solution is next heated to a temperature of 700°C in a muffle furnace. In around five minutes, the entire combustion process was finished. The mixed solution quickly experiences thermal dehydration, releasing gaseous products, growing silicates, and igniting to create a self-propagating flame. After a short while, the crucible is removed from the furnace and left outside to cool. When the phosphor has cooled, it takes on a fluffy appearance, which is subsequently ground with the use of an agate mortar and pestle. The final product obtained was post-annealed at 970°C for 1 hour. Obtained final white powder sample (Fig: 2) was restored in airtight bottle for further FTIR characterization studies.

The chemical reaction of this process is given as follows:

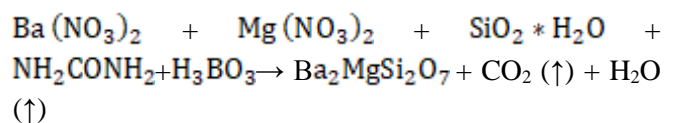


Fig 2: Synthesized Pure $\text{Ba}_2\text{MgSi}_2\text{O}_7$ Sample

3.2 Sample Characterization

This synthesized phosphor's actual formation was discovered using FTIR. With the aid of Bruker Alpha Fourier Transform Spectroscopy, an FTIR spectrum was captured. By combining the sample with analytical-grade potassium bromide (KBr) with pallet preparation, it is possible to investigate the functional groups (4000 to 1400 cm^{-1}) as well as the finger print area (1400 to 400 cm^{-1}) of synthetic phosphor.

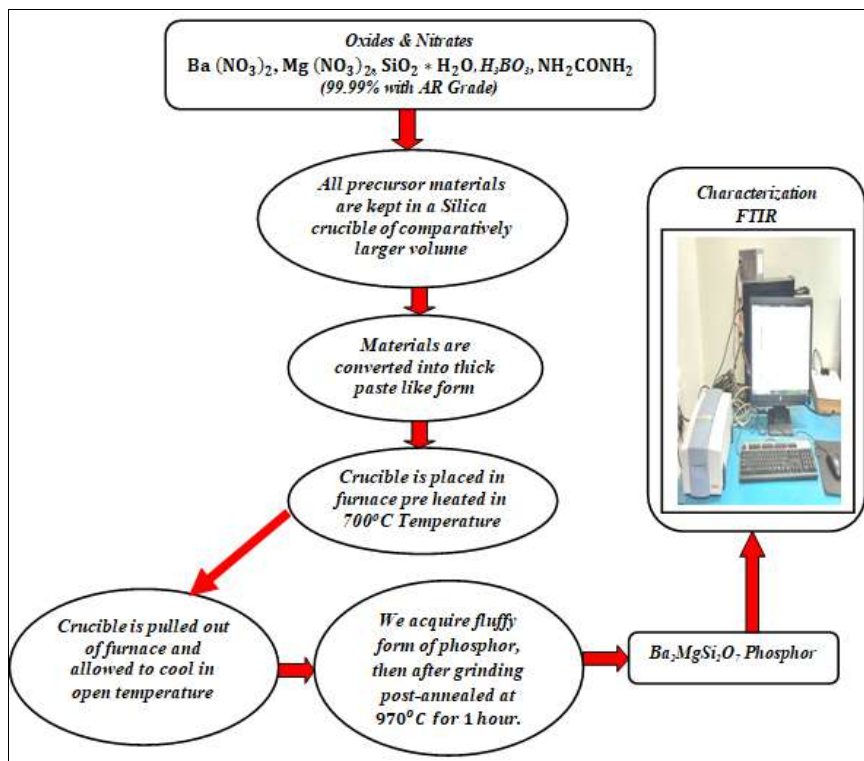


Fig 3: Experimental Procedure

4. Theoretical Framework of FTIR Spectra

A molecule's atoms vibrate continuously one on each side of their overall average since they do not exist in a fixed proportionate position. Such an oscillation mode is present in the IR zone if the periodic change in charge distribution results from vibrational motion. The electromagnetic spectrum's infrared region covers wavelengths of 100 m to 1 m. On this basis, we can observe that the oscillating molecules simply absorb energy from those radiations, with which they can successively interact. In other words, we can say that they are produced by radiation that vibrates at a frequency all its own. This causes all functional clusters to have unique vibrational repeats that are overly sensitive to the surrounding species and its chemical environment. The presence or absence of a particular vibrational frequency in the infrared spectra of any molecule provides important information about how that unique molecule was created.

The mathematical form of the vibrational frequency is:

$$\nu = 2\pi k\mu$$

k stands for [force constant] and μ [reduced mass] respectively. Stretching and bending are two related processes that take place in the bonds tying together the atoms in a molecule. The IR spectrum [FTIR spectroscopy] is used to learn about the molecular production of seemingly all kinds of specimens in any substantial phase, such as solid, liquid, or gas. As a result, those absorb IR radiation.

4.1 Applications of FTIR Spectroscopy

The use of this FTIR technology is widely used, and it is notably preferred in the manufacturing industries of polymers, pharmaceuticals, medical devices, food, and chemicals. The IR spectrum is linked to molecule vibrations that are unique to each drug, just like a fingerprint is necessary for a specific person. With little to no specimen preparation, it is possible to measure IR microscopes with amplitudes up to 10 μ m.

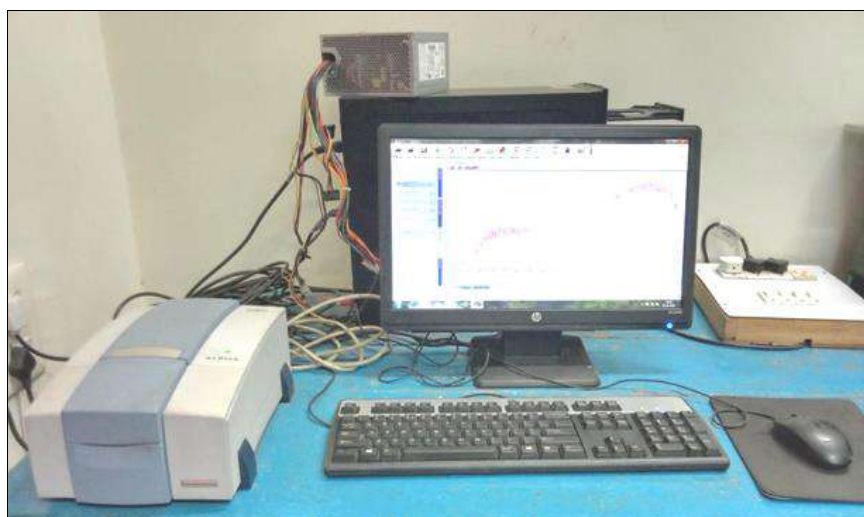


Fig 4: Bruker Alpha Ftir Spectrometer

4.2 Explanation of Spectra

The acquisition of IR spectra has advanced significantly, and it is used in FTIR. To identify a chemical compound's functional groups, additional important equipment called FTIR is used. Inquiries into specific elements of an unidentified blend can also be made using its unique features. Infrared light also causes a chemical molecule to vibrate when seen in this way. This situation results in an interaction between the molecule and the electrical zone of radiation. The dimension of the vibrational motion is changed as a result. In this perusal, spectra were accumulated in the region between ($4000 - 400 \text{ cm}^{-1}$). Nowadays especially, computerised FTIR equipment is

often used. They therefore show to be more effective and sensitive instruments than the traditional dispersive ones. In this investigation, a BRUKER ALPHA FTIR spectrometer (Fig. 4) was employed. When studying an organic chemical in depth, the IR region between $4000-400 \text{ cm}^{-1}$ is significant. However, we are aware that the IR spectra show the emergence of several bands. Since optical isomers are the only other substances that have an identical IR spectrum, any two compounds do not. In the meantime, we see that the IR spectra may also be thought of as the molecule's unique fingerprint. Usually, we examine an IR spectrum in terms of two zones.

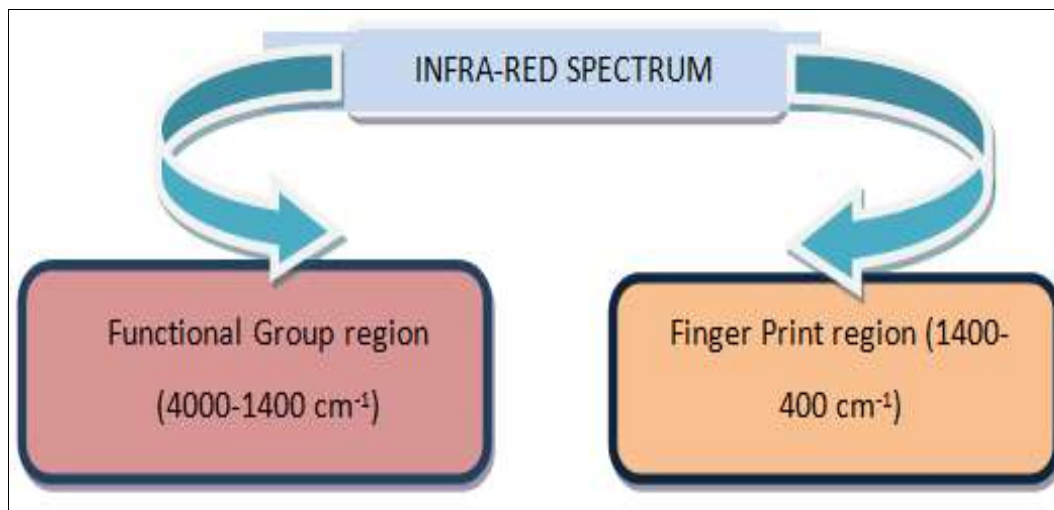


Fig 5: Types of IR Spectrum

[A] Functional Group Region

Functional group area refers to the high frequency range between $4000-400 \text{ cm}^{-1}$. Strong absorption bands that form during stretching oscillations between hydrogen and specific other atoms with a mass of nineteen or less are often present in the zone, which is mostly between $4000-2500 \text{ cm}^{-1}$.

[B] Finger Print Region

The region is referred to as the finger print region and is primarily between $1400-400 \text{ cm}^{-1}$. This is mostly due to the fact that the absorption pattern in this region seems to be unparalleled for a particular molecule. Basically, this region of the spectrum ($1300-909 \text{ cm}^{-1}$) is quite complex. Because this process, the absorption in this zone is produced by both the stretching and the bending modes. The IR spectrum's patterns are the most intelligent in this region, when molecules undergo stereo chemical or hackneyed chemical changes.

5. Results and Discussion

FTIR Spectra

▪ Potassium Bromide (KBr) Pallet Preparation

The $\text{Ba}_2\text{MgSi}_2\text{O}_7$ phosphor is included in the potassium bromide (KBr) pallet. It is vitally important to combine and

crush a synthetic sample with KBr (IR Grade) powder before taking its FTIR spectrum. After apply to hydraulic pressure to create a thin pallet. It is crucial to remember that extremely little amounts of the KBr powder and $\text{Ba}_2\text{MgSi}_2\text{O}_7$ sample should be consumed. In order to make the pallet thinner. FTIR readings and spectra are very clearly captured in this manner.

▪ FTIR Spectra of $\text{Ba}_2\text{MgSi}_2\text{O}_7$ Phosphor

For the purpose of gathering the IR [infra-red] absorption spectrum of the phosphors, FTIR is a non-damaging, simple, and molecular-spectroscopic technological technique. The FTIR spectrum of this $\text{Ba}_2\text{MgSi}_2\text{O}_7$ Phosphor has been showed in fig: 6. This was recorded in the range of (4000 cm^{-1} to 400 cm^{-1}). The bands have centred at 456.37 , 587.49 , 672.35 , 843.67 , 956.64 and 1055.35 cm^{-1} can be allocated owing to the existence of SiO_4 functional group. In addition, considering the absorption bands, validated at 672.35 cm^{-1} and 587.49 cm^{-1} , respectively could be responsible to the existence of SiO_4 group. Thus, the absorption bands of silicate groups were clearly evident in the (IR) infra-red spectrum.

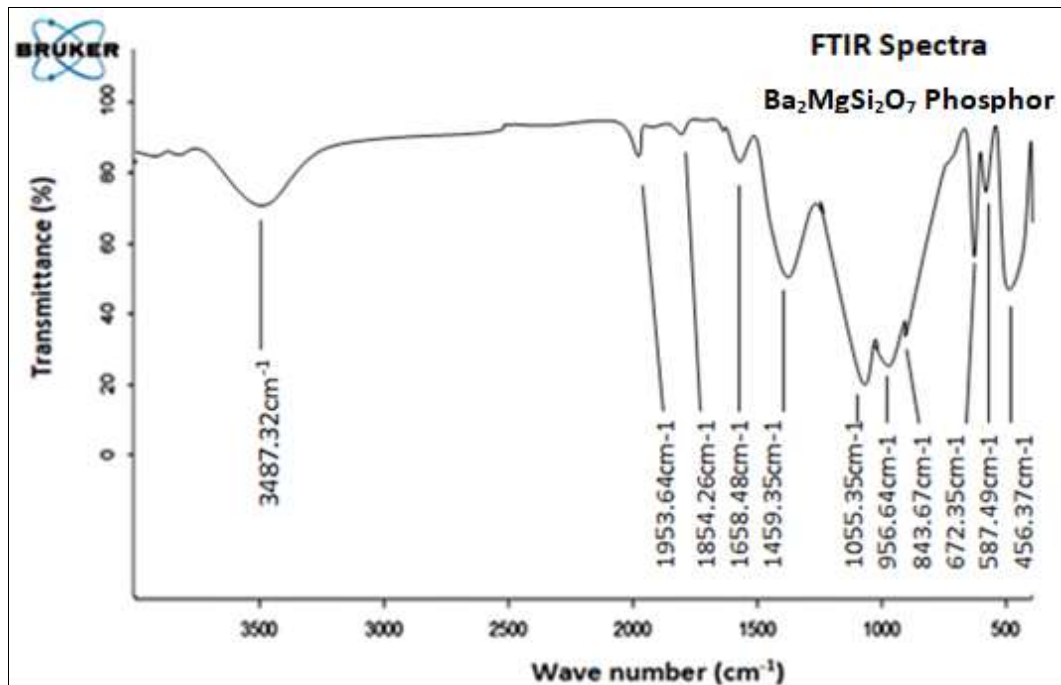


Fig 6: FTIR Spectra of Synthesized $\text{Ba}_2\text{MgSi}_2\text{O}_7$ Phosphor

The sharp band centred at 843.67 , 956.64 and 1055.35cm^{-1} was allocated to the Si-O-Si asymmetric stretching. The bands at 672.35cm^{-1} may be responsible to the Si-O symmetric stretch and Ba-O bending vibrations. The bands bending at 587.49 and 456.37cm^{-1} are allocated to the Si-O-Si vibrational mode. The Peak centred at 843.67cm^{-1} may be responsible to Mg-O bending vibrations. The free ion has a D_{3h} symmetry 69 (trigonal planar) and the band (asymmetric stretching) on its spectrum dominates at 1658.48cm^{-1} . The bands centred at 1854.64 and 1953.64cm^{-1} is responsible due to carbonation reaction mechanism. This can be lead to distortion in the lattice resulting in 1459.35 and 1658.48cm^{-1} vibration modes allocated to vibration in divalent barium ion [Ba^{2+}] and divalent magnesium ion [Mg^{2+}] respectively. Various peaks in the range of 1854.26 and 1953.64cm^{-1} are possibly due to the presence of Ba-O stretching. The peak centred at 3487.32cm^{-1} is visible due to O-H stretching. This signs that the presence of moisture in the prepared powder sample.

6. Conclusion

In summary, $\text{Ba}_2\text{MgSi}_2\text{O}_7$ powder sample was successfully synthesized via low temperature traditional combustion synthesis technique. Prepared powder sample was optically characterized by Fourier Transform Infra-Red Spectroscopy (FTIR) also reported. The FTIR (Fourier Transform Infra-Red) spectroscopic analysis has been extensively utilized for the investigation of organic & inorganic compounds and identification of absorption bands as well as different functional groups which is clearly present in spectra. The silicate (SiO_4) absorption bands and different functional groups of related pure compound to well agreement for actual clarifications have been examined under the FTIR spectra.

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also thankful to kind support NIT Raipur (C.G.) India for the facility of FTIR Spectroscopy.

8. Conflict of Interest

The authors declare that no any conflict or any economic interest that exists in our present research work.

9. Authors Contributions

This work was carried out in collaboration among all authors. Author Dr. Shashank Sharma undertakes the manuscript designed and conducted the entire experiments & characterization studies, collected and analyzed the research data, and prepared the entire manuscript draft as well as supervised the results-discussion. Similarly, author Dr. Sanjay Kumar Dubey has properly checked the spelling mistake, punctuation, grammatical error, conceptualization, writing, review, editing and helped in sample preparation. All authors read and approved the final manuscript.

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