Influence of Potassium Chloride Doping on Carbamide Butanedioic Acid

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ABSTRACT

Potassium Chloride (KCl) as an additive is added into Carbamide Butanedioic Acid by the method of slow evaporation solution growth technique at room temperature to get a new crystal. The grown crystals have been subjected to different instrumentation methods. The presence of functional group for the grown crystals has been identified by FTIR analysis. UV-Vis, TG/DTA and Vickers hardness methods assessed the optical, thermal and mechanical behaviors. Agar well diffusion method showed the antibacterial effect of CBA doped KCl on different microorganisms

1. INTRODUCTION

Since the present clinical path is still insufficient to address the issue of the rising appearance and spread of antibiotic resistance, the quest for novel active antibacterial agents is gaining importance. In the medical field, there is a pressing need for the creation of novel antimicrobial agents. For the treatment of cancer, malaria, and neurological illnesses, various metal-based complexes (based on silver, copper, iron, gold, bismuth, gallium, etc.) have been developed over the past 20 years and have entered human clinical trials (Biot, C et al 2011, Monro at al, S 2019, Kenny, R.G. and Marmion, C.J. 2019). More than a few classes of antibacteiral compounds are at the moment existing microorganism's resistance to these drugs continuously rising. With the purpose of avoid this serious health trouble, the embellishment of novel types of antibacterial agents or the development of bioactivity of the unsurprisingly known biological active compounds is a very interesting research problem. It is well known that Carbamide and Butanedioic Acid derivatives possess a variety of biological activities. They shows broad spectrum of pharmacological effects. Several compounds containing Carbamide and Butanedioic Acid have been reported to exhibit biological activities. Moreover, CBA doped KCl behaves as an antibacterial agent which inceases the activity. In the present work, KCl doped CBA crystal have been successfully elaborated. The structural, spectral, mechanical and optical properties of this crystal was investigated. The doping effects on KCl single crystal was observed and highlighted.

2. EXPERIMENTAL DETAILS

Carbamide and Butanedioic Acid were combined in a 1:1 molar ratio to create the potassium chloride (KCl) doping Carbamide Butanedioic Acid combination, which was then dissolved in deionized water. Next slowly add potassium chloride dopant with a 2 M concentration. The mixture was continually stirred with a magnetic stirrer for six hours before filtering. The remedy might be used at room temperature. A colourless transparent crystal was collected after three weeks of growth, and the picture of the CBA crystals that had been formed and been doped with KCl is displayed in Figure 1.

Fig. 1 Picture of the CBA crystals doped with KCl

Using a Reich seifert diffractometer with K radiation (λ= 1.5418), the powder XRD pattern of the formed CBA crystal was recorded. The FT-IR spectra of TGP have been acquired using a Bruker IFS 66 V Spectrometer in the range 4000-100 cm^{-1}. Energy Dispersive X-ray studies were performed using a Bruker EDS spectrometer in conjunction with the SEM. The FTIR spectrum was captured between 400 and 4000 cm^{-1} using a Perkin-Elmer FTIR spectrometer and the KBr pellet technique. UV-vis spectrum was captured using a Lambda 35 Spectrometer between 190 and 1200 nm. In order to understand thermal behaviour, thermal analysis was done simultaneously using a Perkin Elmer thermogravimetric and differential analyser (Mode: PYRIS DIAMOND) in nitrogen atmosphere heated from 400 to 800 at a heating rate of 100°C. Using the HIOKI 3532 LCRHITESTER, a dielectric investigation of the formed crystal was conducted at various frequencies and temperatures. Vicker's micro hardness indentor was used to conduct the microhardness analysis. Q-Swiched Nd:YAG High Power Laser (QUANTA RAY MODEL).

3. RESULTS AND DISCUSSION

3.1 PXRD Analysis

To determine their crystallographic structure, CBA crystals were KCl doped. A pure KCl single crystal's PXRD diagram, shown in Figure 2, verified the crystal's high degree of crystalline clarity and phase purity. The crystal system is orthorhombic because the lattice parameters (A=42.1559, B=12.0893, C=8.9269, α=90, β=90, γ=90), are all 90° (a ≠ b ≠ c, α = β = γ= 90°).

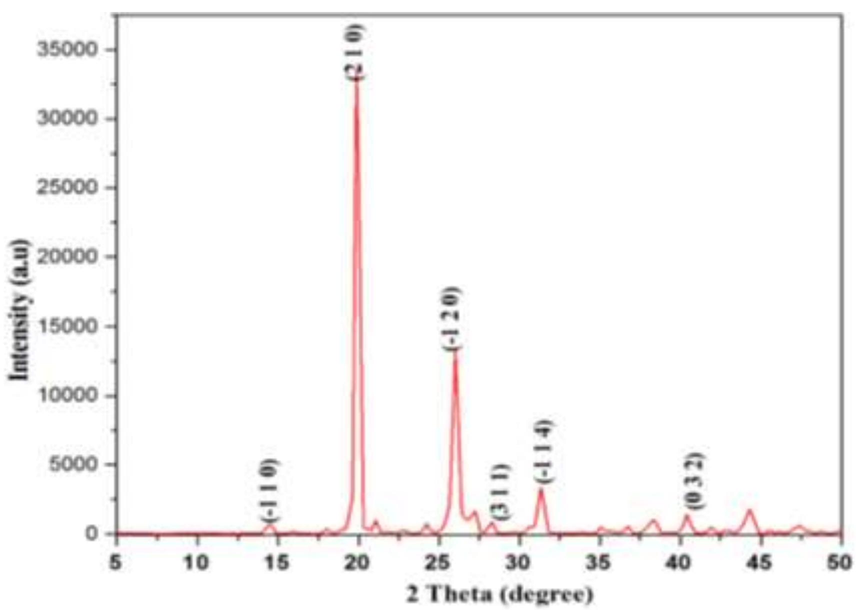

Fig. 2 PXRD of CBA crystal were KCl doped

3.2 SEM and Energy Dispersive X-Ray Analysis

Figure 3 displays the SEM surface of developed KCl-doped CBA crystals. The investigation of the influence of the KCl doped CBA crystals. The surface of crystal faces reveals the formation of structure defect centers (Figure 3). In the presence of KCl doped CBA in the growth medium, the SEM photograph of ADP crystal shows a fibrous structure. Scatter centers are observed in KCl doped CBA

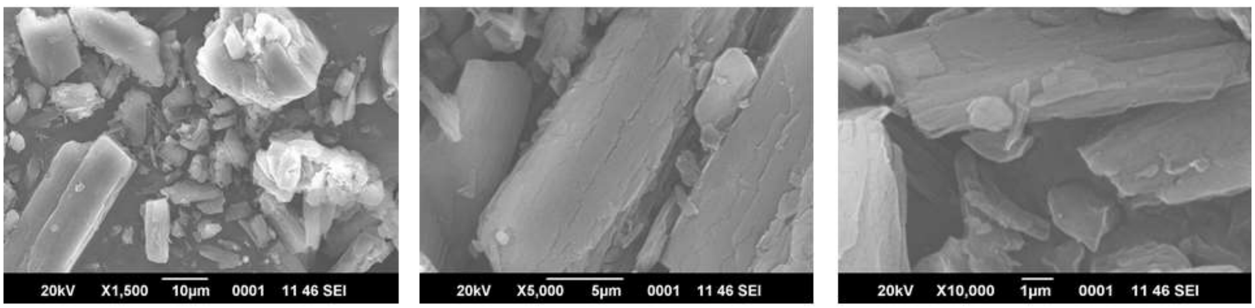

Fig 3 SEM Analysis

Figure 4 displays the spectra from the EDAX analysis that display the purity and chemical makeup of the produced crystals. Figure 3's formed crystal of potassium chloride, which is pure according to the spectrum, is visible.

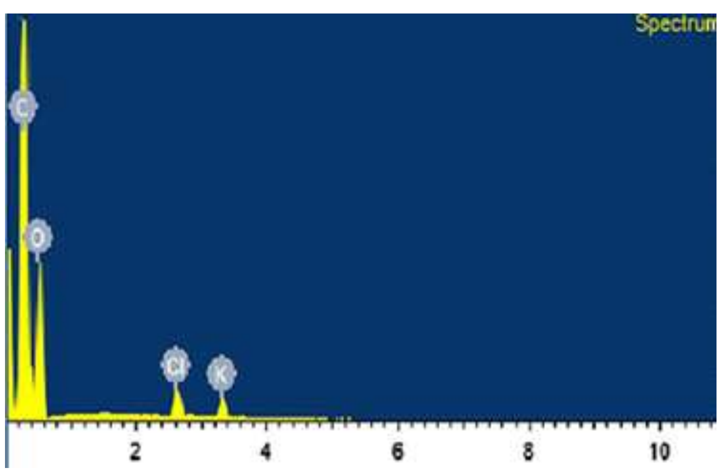

Fig 4 Energy Dispersive X-ray Analyses (EDAX) spectrum

The energy dispersive X-ray analyses (EDAX) spectrum (Fig. 4) indicating the presence of K, Cl, Cand O elements and no other elements were detected, which demonstrates that the CBA doped KCl: crystal has a very high purity. However, the average atomic percentage (Table 1) shows that the crystal is rich in carbon.

Table 1 Elemental analysis of KCl added CBA Crystal

Element content	Line Type	Mass %	Atom %
C	K series	51.07	54.3
O	K series	48.1	45.28
Cl	K series	0.42	0.27
K	K series	0.41	0.15
Total		100	100

3.3 FTIR Analysis

Figure 5 shows the FT-IR spectrum of a CBA crystal doped with KCL crystal and the vibrational assignments are given in Table 2. The finger print region of the IR spectrum is defined as 1418 to 500 cm^{-1} (Vijayalakshmi et al 2021).

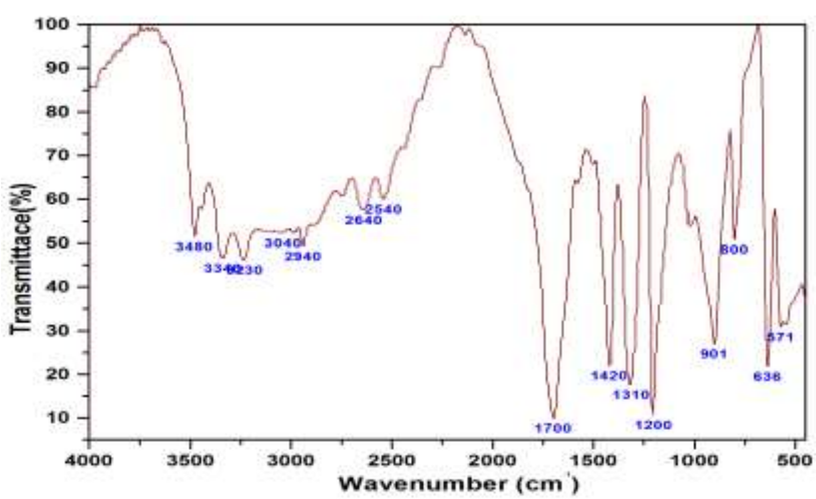

Fig 5 FTIR spectrum

In the finger print region, the potassium, vibration is observed at 571 and 507 cm^{-1}. Presence of these peaks in the doped KCL spectrum clearly indicates the presence of KCL in the lattice of CBA crystal.

Table 2 FTIR band assignments of CBA crystal doped with KCL

Frequency (cm^{-1})	Assignments
3480s	$\nu_{as}NH_2$
3340s	ν O- H
3230s	$\nu_{ss}NH_2$
3040m	ν CH
2940m	ν CH
1700vvs	ν C=O
1652w	βOH, δNH2, τCN
1420vs	τNH, τCN, βO-H
1310vs	βOH, $\nu_{as}CO_2$
1200	τCN, βOH, ρNH_2, ρNH_2
901s	νCC, βCC, νC-N
800m	ωNH, ωNH_2, τCN, τNH, δCO2
636vs	τOH, ωCO_2, δCO2
571vs	$\rho 2CO_2$, ρCO_2, ωCO_2, ρCOO-
506w	ωCO_2, ρCH_2, τOH

υ_{SS}- symmetric stretching; υ_{AS}- asymmetric stretching; υ_{IP}- in-plane stretching; υ_{OP}- out of plane stretching; β-in-plane bending; ω- wagging; δ- scissoring; τ-torsion; ρ-rocking;

In the crystals' functional groups for generated KCL mixed CBA crystal, the spectrum is displayed in Fig. 5. A highly challenging area to assign absorption bands to is the finger print region. NH_2 and C=O stretching vibrations are shifted from CBA which strongly evidences of inter molecular interactions when KCL mixed CBA crystal. Its absence from the pure CBA spectrum and its appearance in the doped CBA spectrum both strongly suggest that the CBA crystal's lattice contains an KCL

3.4 UV-Visible analysis

The recorded UV spectrum of CBA doped with KCl is shown in Fig. 6. The lower cut off region is obtained at 215 nm and there is a steady transmittance in the visible region from 200 to 800 nm and the band gap is 5.7eV. This wide range transparency of this crystal is an added advantage in the field of optoelectronic applications.

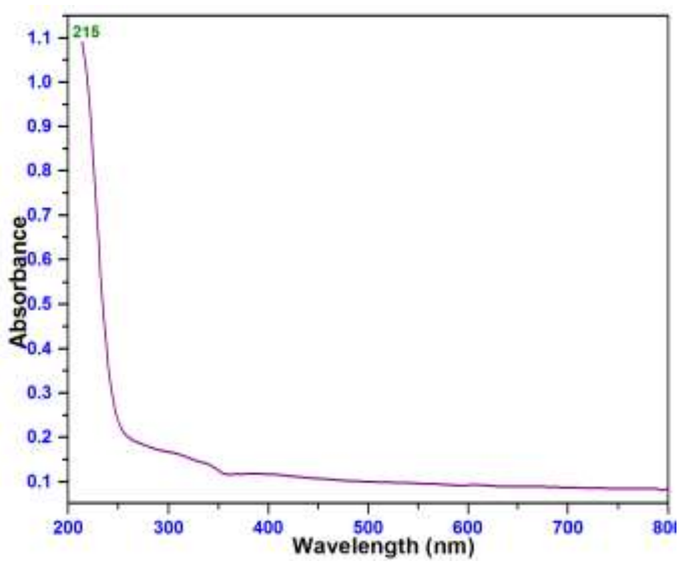

Fig. 6 UV-visible absorption spectrum of CBA crystals doped with KCL

3.5 Thermal Analysis

TG/DTA is used to identify the thermal stability of the grown crystal. TG/DTA patterns obtained in the present work are shown in figure 7. The thermogravimetric analysis of KCl doped CBA was carried out and the obtained thermogram is shown in Fig. 8.6. At 247°C, there is a noticeable weight loss. Below 247 °C, water starts to evaporate, demonstrating the presence of absorbed water in the crystal lattice. The melting point of the crystal is set at 247 °C, where there is a fast weight loss without any intermediary stages. According to this study, the chemical may be utilised for device manufacture below the crystal's melting point. The material appears to undergo an irreversible exothermic transition between 163 and 289 °C, according to the DTA curve measured for the formed crystal. A prominent exothermic peak at 242 °C indicates the crystal's melting temperature. The DTA thermogram also shows that the CBA's steep exothermic peak coincides with it, confirming the material's thermal stability of the crystal.

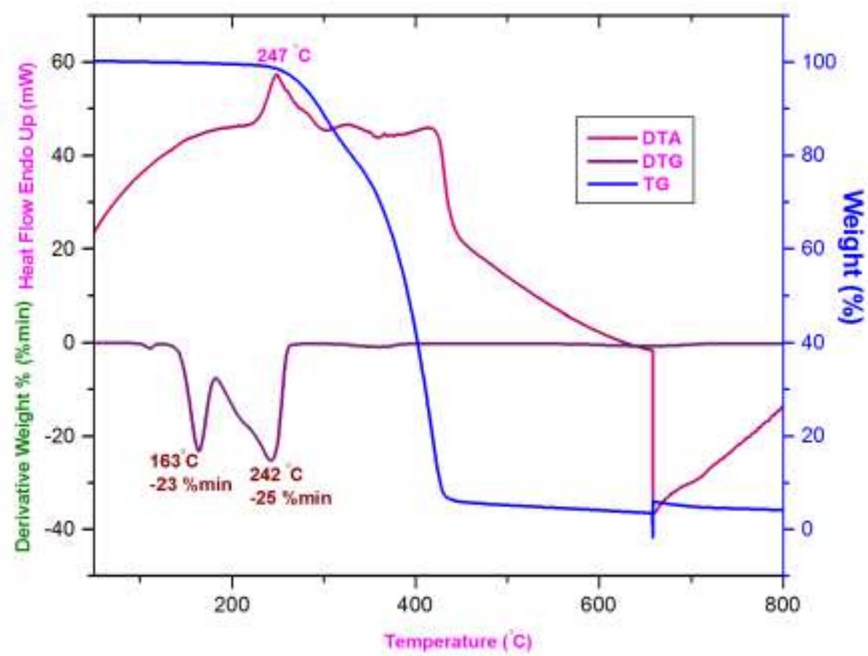

Fig 7 Thermogravimetric analysis

3.6 Mechanical study

When compared to the findings of the prior study, the results of the present study's micro hardness analysis demonstrate higher mechanical quality. According to the current work, the chloride absorption into the CBA lattice probably strengthens the interaction with the host molecules, which is why the hardness value of the KCl-admixed CBA crystal increased.

The values for the (n) were determined using the least square fit method, and they are shown in Figure 8. From the calculated work hardening coefficient grown crystals belong to the category of hard materials because the KCl admixtured CBA crystal was below 1.6(Onitsch, EM, 1956).

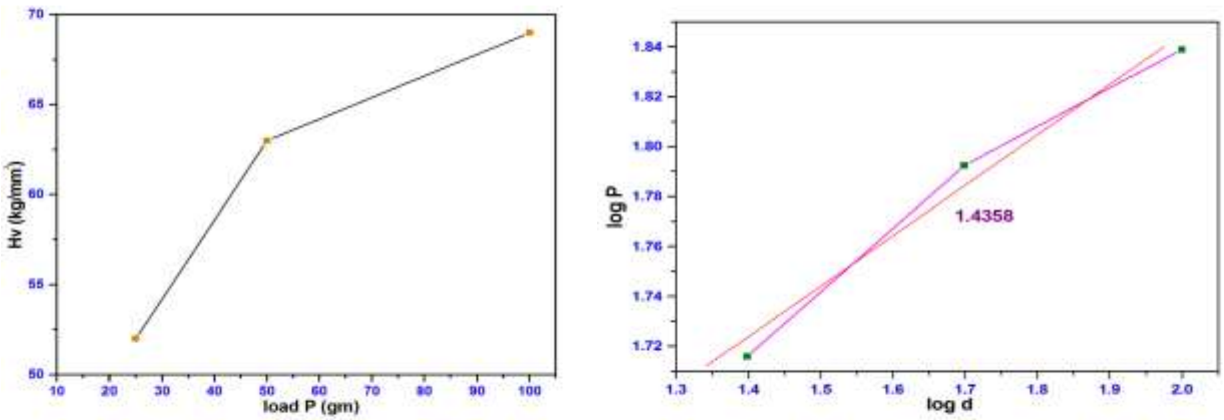

Fig 8 The plot between the hardness number (Hv) and the associated loads (Kg)

3.7 Dielectric study

The expansion of the crystal, electronic and ionic polarisations, as well as the existence of impurities and crystal defects, are all frequently implicated in the variation of the dielectric constant with temperature. Sharp peak at phase transition temperature reveals the quality of generated crystal of continuous phase transition. At the transition temperature of 328 K, the observed value of the dielectric constant reached its maximum. Dielectric loss varied well, as shown in figure 9, but it is greatest at curie temperature and lowest in the paraelectric phase.

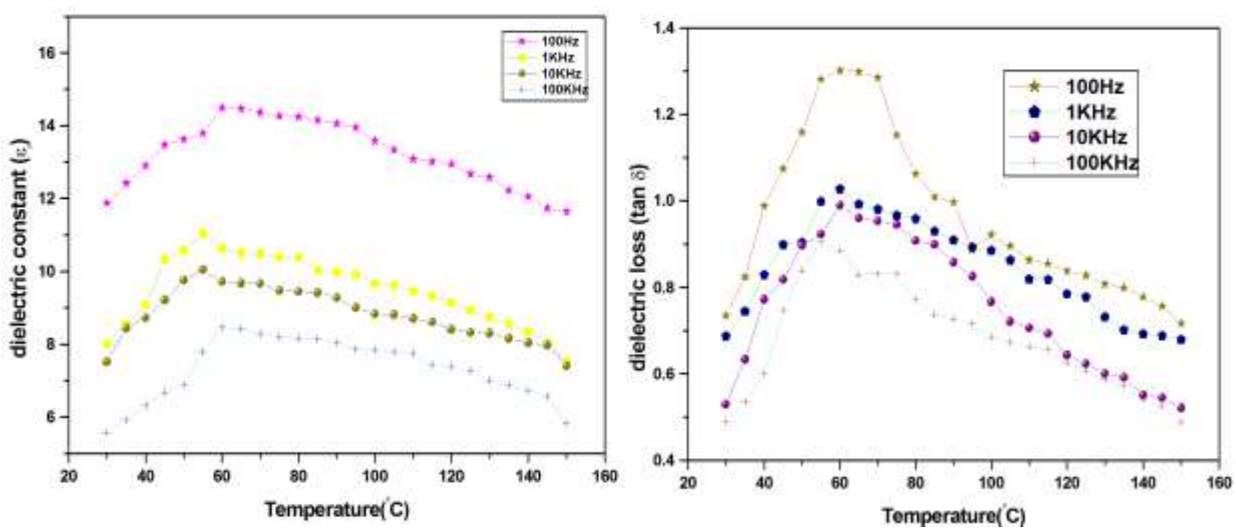

Fig 9 Dielectric study

3.8 Antibacterial assay

Agar well diffusion method is commonly used to check the antibacterial activity of CBA doped KCl. Four microorganisms, namely Bacillus cereus, Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa were used for this purpose.

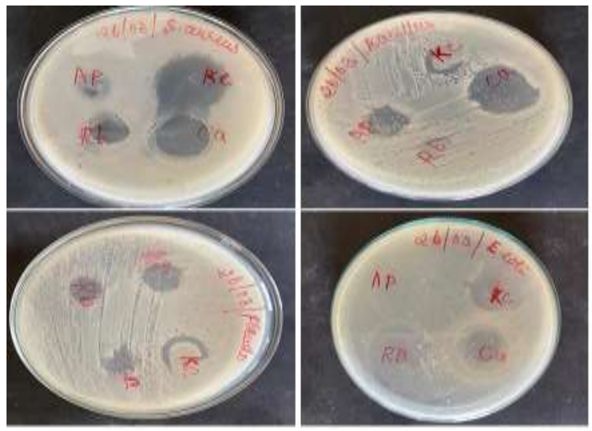

Fig 10 Photographs exhibiting the actions of CBA doped KCL (labeled as KC) against bacterial pathogens

1 mg/ml solution of CBA doped KCl was made Subsequently, wells of 8 mm diameter were punched into the agar medium and filled with 20 µl (25 mg/ml) of sample and allowed to diffuse at room temperature for 2 h. The plates were then incubated in the upright position at 37° for 24 h. After incubation, the diameters of the growth inhibition zones were measured in mm as shown in Fig. 10. After 24 h of incubation, no growth of B. cereus, and P. aeruginosa was seen but Staphylococcus aureus have 15 mm zone was formed (Table 3). From these results CBA doped KCl shows higher antibacterial activity against Gram-negative bacteria.

Table 3 Antibacterial Assay

Sl. No.	Organisms Names / Test samples	Zone of growth inhibition in 'mm' KC
1	*Bacillus cereus*	-
2	*Staphylococcus aureus*	15
3	*Escherichia coli*	8
5	*Pseudomonas aeruginosa*	-

4 CONCLUSION

The CBA doped KCl single crystals were generated using the slow evaporation solution growth method. In order to compare the unit cell properties of CBA-doped KCl crystals, Powder crystal XRD findings were used. These results show a good agreement with the values from the literature. By using FT-IR Spectral investigations, the functional groups of CBA doped KCl crystal were examined. A The optical behaviours are examined using a UV-vis spectrometer, and the results demonstrate that the crystals have extended transparency down to the UV. TG/DTA studies have been used to assess the thermal stability and melting point values of the pure and CBA doped KCl crystals. Calculated CBA doped KCl crystal hardness values were compared to those of pure. Since bacteria cannot grow in media containing CBA doped KCl, the antimicrobial activity results of the viability assay have demonstrated the excellent antibacterial nature of CBA doped KCl. Therefore, it can be inferred from the aforementioned studies that CBA doped KCl is an effective antibacterial agent for treating illnesses and it can also be done to identify the precise active moiety responsible for the biological activity.

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