IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

SELF-ASSEMBLED HYDROGEL FORMATION INDUCED BY BETA-CYCLODEXTRIN AND COMMAN SURFACTANT. (MICELLE – GEL LIKE TRANSITION)

Salma Dal [1], Muzammil kuddushi[1]

1.department of applied science , Parul university, Vadodara.

ABSTARCT

Two components β-cyclodextrin and common surfactant (sodium dodecyl sulphate) are reacted together and form a hydrogel. This hydrogel formation is studied by various method including transmission electron microscopy (TEM), rheological measurement, FT-IR spectroscopy. By this measurement, it is concluded that β-cyclodextrin is act as a host and sodium dodecyl sulphate (SDS) is act as a guest and they are forming an inclusion complex.

Moreover, the properties of hydrogel formed by β-CD and SDS are sensible to external stimuli e.g., temperature. By rising the temperature, the hydrogel converts into solution and by lowering the temperature, the solution is converted into hydrogel. This process is convertible from solution to gel and gel to solution with increase and decrease temperature respectively.

Also, the external stimuli like Ph, hydrogel is sensible for e.g., in acidic Ph the hydrogel start to degrade. This hydrogel is helpful in biomedical application for example; drug delivery system.

KEYWORDS: SDS, β-CD, HYDROGEL, rheology, FT-IR spectroscopy, TEM.

GRAPHICAL ABSTRACT

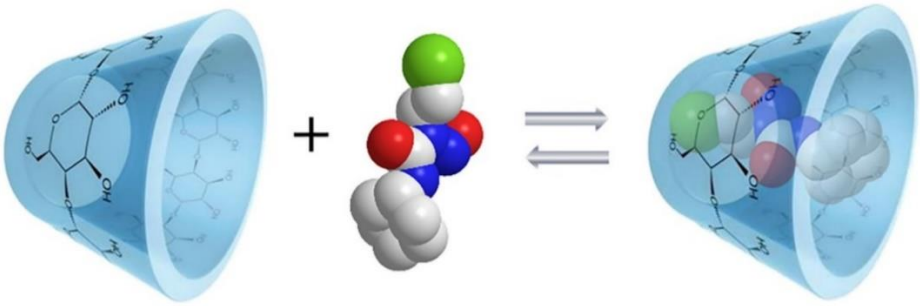

fig 1. Graphical abstract

1. INTRODUCTION

β-CD is a natural amphiphilic molecule which is derived from starch, it has a truncated cone shape with a hydrophobic cavity and hydrophilic exterior. β-cyclodextrin is an oligosacceride which contain 7 glucose unit linked by α-1,4 linkages. cyclodextrin's water soluble nature, non-toxicity and easily avaibility make them extensively used in chemical industry, pharmaceutical industry & food stuff etc.

Amphiphilic molecule has a tendency to form a various structure like vesicle, warm like, micelle, lamellar sheet and 3D network structure that is gel and it make an important approach in various field like cosmetics, removal of toxic dyes, vehicles of drug delivery, In a tissue engineering and for synthesis of nano material.

β-cyclodextrin is a macromolecule which is widely studied in supramolecular chemistry as a host molecule because it has a tendency to trap the guest molecule of any tiny range compound by the hydrogen bonding. the compound may be organic or inorganic. therefore, it can be derived that cyclodextrin are extensively used to tune the characteristics of supramolecular system. for e.g., jinglin shen recorded the formation of complex by addition of cyclodextrin and imidazolium gemini surfactant. jiyang et al recorded the formation of inclusion complexes between cyclodextrin & cetyltrimethylammonium bromide.

now a days, ionic liquids have been used to a greater extent because it has an advantageous property like low vapor pressure, high ionic conductivity, more thermal stability and they are easily recyclable. among them common surfactant like sodium dodecyl sulfate is an amphiphilic molecule which have a hydrophobic tail and hydrophilic head, here sulphate group is attached with carbon chain by polar covalent bond. jinglin shen reported that the inclusion complex formed between β-cd and imidazolium gemini surfactant and it is studied by Fourier transform infrared spectroscopy (FT-IR), transmission electron microscopy (TEM) and X-ray diffraction.

In the present work, we have chosen the β-cyclodextrin as a host molecule and sodium dodecyl sulphate which is a common surfactant as a guest molecule. The interaction between these two components from the three- dimensional network by hydrogen bonding which is gel. the product has been studied by various techniques such as transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), rheological measurement. the result shows that the host guest interaction between two components.

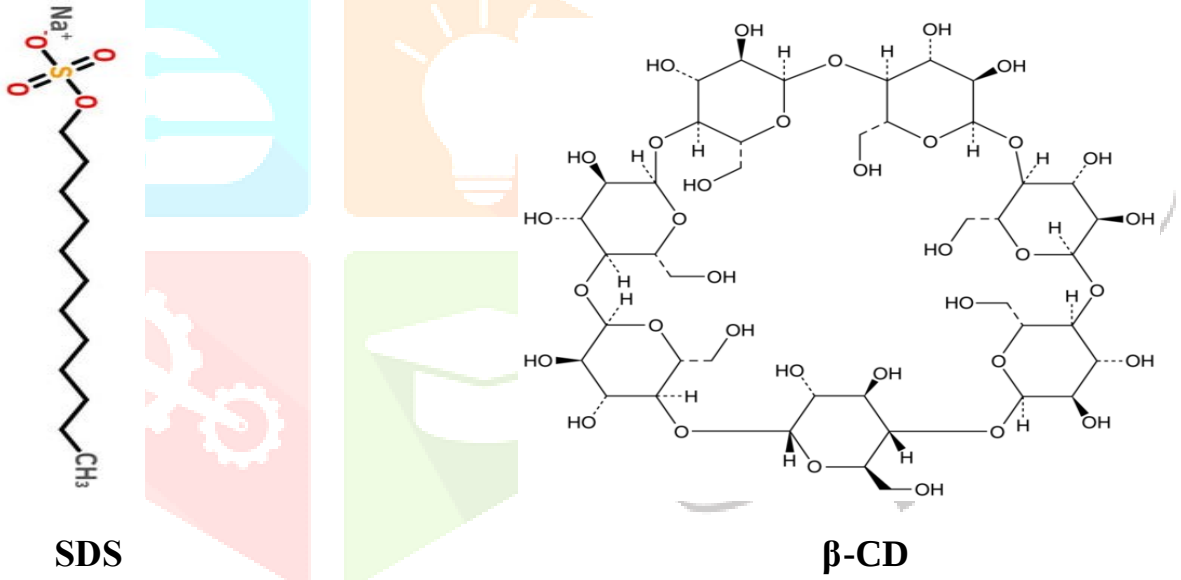

Fig.2 Structure of SDS and β-cd

2. EXPERIMENTAL SECTION:

2.1 MATERIAL

The chemical used in this experiment like β-cyclodextrin which have (≥ 98% purity) was purchased from sulab chemicals, the common surfactant sodium dodecyl sulfate was purchased from oxford lab fine chemicals which have (≥97% purity), water used in this experiment was one-time distilled water.

2.2 PREPARATION OF SOLUTIONS

To obtain a hydrogel , desired amount of amPhiPhiles $NaC_{12}H_{25}SO_4$ sodium dodecyl sulfate and β-cyclodextrin were weighted in the sealed glass vials and suspended in distilled water at various concentration. The mixtures were sonicated for 1 min at 20 KHz (sonics vibracell) until homogeneous mixture was obtained prior to the measurements, all the solutions were kept for 7 days at room temperature to maintain the equilibrium.figure 3, indicate the various step during increasing the amount of β-cd in SDS that how the solution transform from solution to viscous , viscous to turbid ,turbid to opaque gel.

Fig 3. Hydrogel formation with increasing concentration of β-cd in SDS

2.3 METHOD AND CHARACTERIZATION

2.3.1 Phase behavior: it can be seen by visually. here, as concentration of β-cd increases in SDS, gel formation occurs but with temperature increases gel degrade into solution to certain concentration.

2.3.2 TEM observation: TEM images of hydrogel is carried out on a Philips CM200 electron microscope at a working voltage of 200 kv. A small amount of sample was placed on a carbon coated copper grid, then copper grid was freeze drying in a vacuum extractor at -60˚for one day.

2.3.3 Rheological measurement: rheology is a key method to investigate gels, from which viscoelastic properties of gel can be discovered. here, rheometer Physics MCR 301 with a plate-plate geometry of 49.973 mm diameter and default gap of 0.4mm was used. these measurements were done within the viscoelastic region where G' and G" are independent of strain amplitude.

2.3.4 FT-IR spectroscopy measurement: FT-IR spectra of SDS and hydrogel forming by common surfactant (SDS) and β-cd are measured on a FT-IR-8400 spectrometer with KBr pallets in a 400-4000 nm region.by carefully investigation of the gel's self-assembly mechanism, the location of functional group indicates the interaction between the molecule -molecule and molecule -solvents. The FT-IR spectra of SDS and hydrogel is shown in figure 8.

3. RESULTS

3.1 Preparation of hydrogel: in Sodium dodecyl sulphate solution, we added β-cyclodextrin. figure 4, suggest that after certain amount of adding β-cd the opaque gel is form. which indicate that the compact structure between β-CD & SDS is formed. it suggests that the hydrogel is formed by some chemical interaction between these two components. this hydrogel is further studied by various methods.

Figure 4. preparation of hydrogel

3.2 Phase behavior of β-cd/common surfactant mixed system: first the Phase behavior of β-cyclodextrin / sodium dodecyl sulphate mixed system is observed. Figure.5 shows the Phase diagram of β-cd/SDS mixed system obtained after equilibrated at 20.0±0.1˚ for at least one weeks. By changing the concentration of β- cyclodextrin in sodium dodecyl sulphate, we get a different state of product which is turbid - viscous - gel. by rising the concentration of β-cd in SDS, the Phase diagram shows the transformation of solution to turbid, turbid to viscous, viscous to gel. Figure.3 shows the Photo of seven different sample of mixed component by increasing the amount of β-cd in common surfactant, the sample transform from sol to gel which is seen in Photo. It suggests that the gelation is occur by host – guest complex formation.

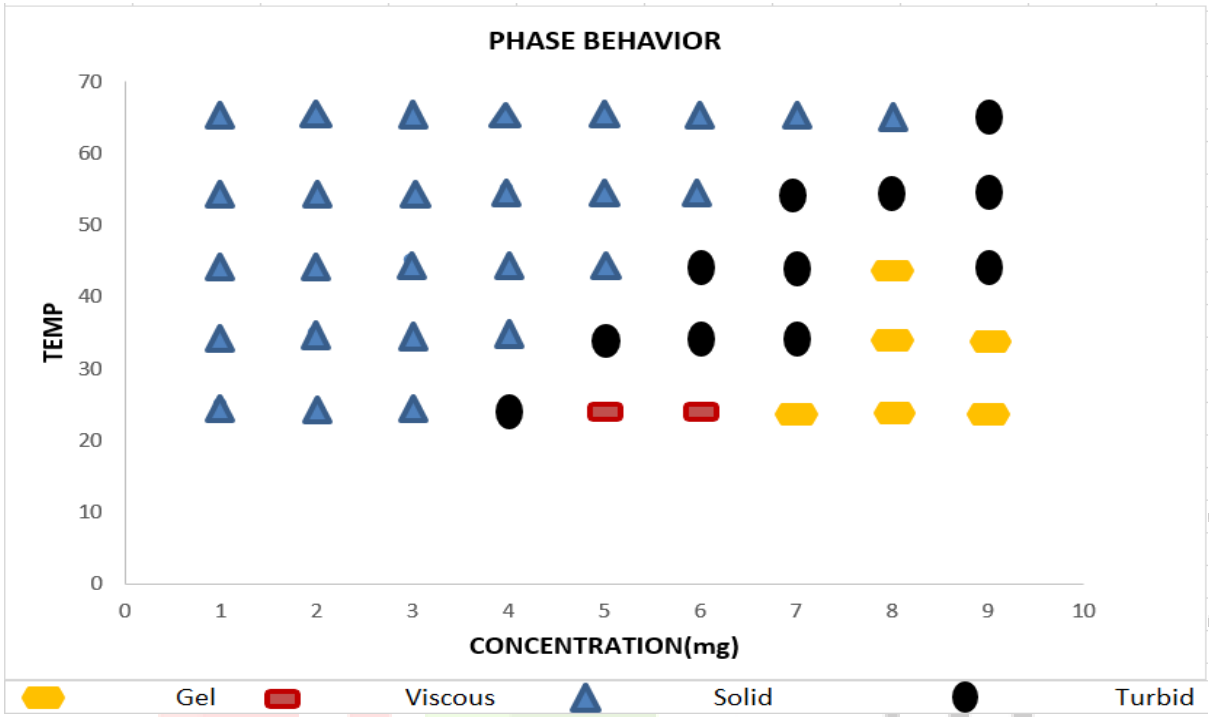

Fig 5. Phase behavior of hydrogel (conc vs temp)

3.3 degradation behavior: the degradation behavior is seen in figure 6. it indicates that at acidic Ph(5Ph) by adding a small amount of acid in hydrogel the degradation with respect to time is fast while in basic Ph (7.4Ph) the degradation rate with time is quite small compare to acidic Ph. This result indicates the nature of hydrogel is sensitive to external stimuli like Ph.

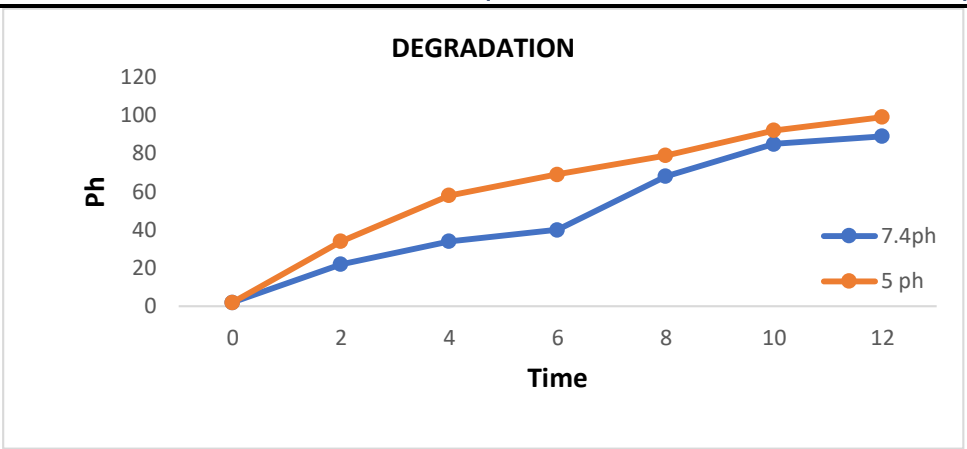

Fig 6. Degradation behavior (time with Ph)

3.4 rheological measurement: rheology is a key method to investigate the gel, from which the mechanical and viscoelastic property of the gels can be extracted. figure 7 indicate the rheological property of gel containing different concentration of β-cd in SDS. Figure 6. Indicate the storage modulus (G') and loss modulus (G") of two component hydrogel within a viscoelastic region. Where G' and G" are independent from applied stress. The blue line denotes the value of stress modulus (G') and the orange one denotes the value of loss modulus(G").

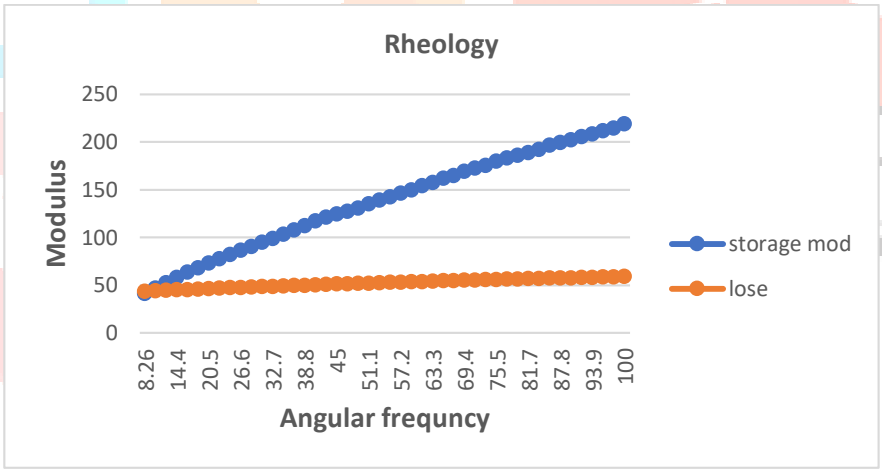

Figure 7. Rheological measurement

3.5 FT-IR results: The FT-IR spectra of SDS and β-cd / SDS hydrogel are shown in figure 7. Better understand of the gelation mechanism, we perform FT-IR study to understand the mechanism of the gelation in details. Here we can see in figure 7, frequency of the -OH group of β-CD and -S-O frequency of cationic head group of ionic liquid were shifted to lower wavenumber due to the formation of the hydrogen bonding. After the formation of the gel, -CH frequency of gel state were shifted to the red shift due to the stronger hydrophobic interactions. Finally, we concluded that hydrogen bonding and hydrophobic internecions which play the major role during the gelation state.

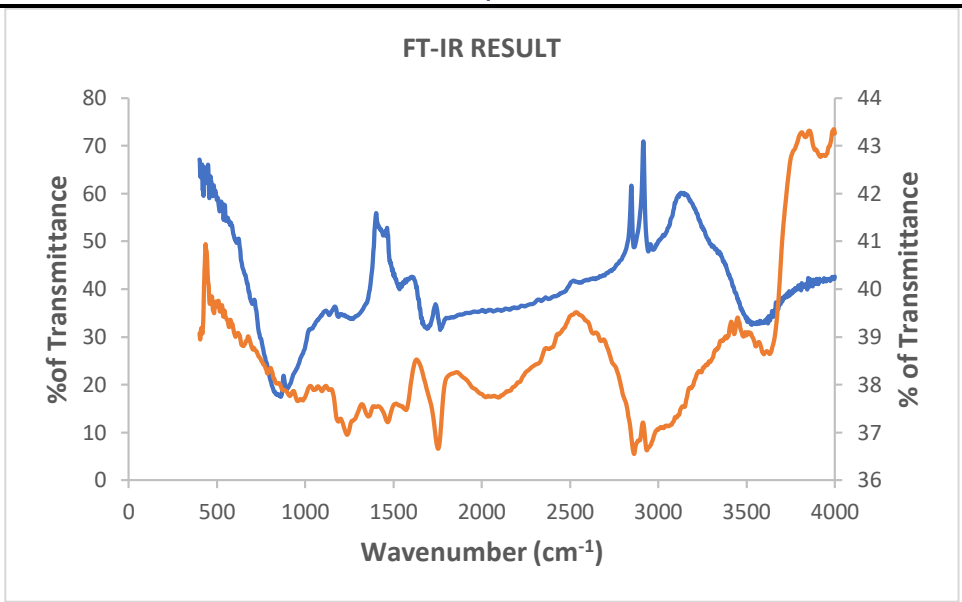

Figure 8: FT-IR spectra of hydrogel

3.6 TEM results: the hydrogel morphology was further examined through transmission electron microscopy (TEM), a small amount of sample was placed on carbon coated copper grid, then copper grid is freeze drying in vacuum extractor at -60˚C for 1 day. we observed in figure 8, the images of TEM indicate the chemical interaction (hydrogen bonding) and formation of fibrous 3D network at different magnification like 0nm ,5nm and at 10nm.

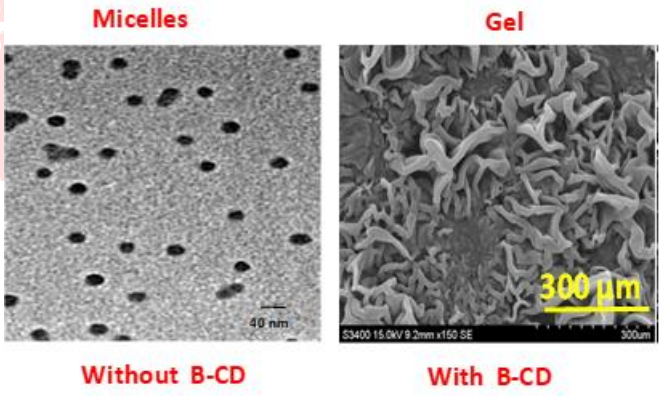

Figure 9. TEM image of hydrogel

FIGURE DETAIL

Figure 1. graphical abstract.

Figure 2. structure of sodium dodecyl sulphate and β-cyclodextrin.

Figure 3. hydrogel formation with increasing concentration of β-cd in common surfactant. gel formation is seen from left to right as oligosacceride concentration increases.

Figure 4. preparation of hydrogel.

Figure 5. Phase behavior of β-cd/sodium dodecyl sulphate with increasing concentration and temperature for at least 3 weeks.

Figure 6. degradation behavior is clearly seen by temperature vs two different Ph at certain time.

Figure 7. rheological result of hydrogel that shows viscoelastic property of inclusion complex. (A) elastic modulus(G') and (B) viscous modulus (G") as a function of the applied stress at varied frequency

Figure 8. FT-IR spectra of hydrogel sample

Figure 9. TEM images of hydrogel sample which formed by β-cd and sodium dodecyl sulphate.

CONCLUSION

In conclusion, β-cyclodextrin /sodium dodecyl sulphate hydrogel is subsequently prepared in this experiment and their character were investigated effectively by FT-IR spectroscopy, transmission electron microscopy (TEM) & rheological measurement. The result indicate that the sol-gel transformation occurs by increasing the ratio of β-cyclodextrin in common surfactant which is sodium dodecyl sulphate. Moreover, by rising temperature the hydrogel of β-cd /SDS can be changed to solution because degradation of gel occurs. This process is reversible and reproducible. Mechanism of this gel is that hydrophobic part of sodium dodecyl sulphate is trap into cavity of β-cyclodextrin which is supramolecule. Here, hydrogen interaction between β-cd and SDS happen. Which result to the formation of hydrogel. Due to their strong mechanical property, this hydrogel can be widely used in drug delivery system.

REFERENCES

1. Jinglin Shen Lifie song xia xin Dan wu Shubin wang rui chen guiying xu j colsurfa (2016) self-assembled supramolecular hydrogel induced by β-cyclodextrin and ionic type imidazolium gemini surfactant.
2. M.kuddushi , Arvind kumar , debes roy vinod kumar, ACS omega (2020)
3. E. Larrenta .J.R Isasi, non-covalent hydrogel of cyclodextrin & poloxamines for control realese of proteins (2014)
4. A Harada, Y.Takashima , H. Yamaguchi , cyclodextrin based supramolecular polymers. chem society.(2009)
5. Le Xin song, Hai ming wang , Xue Qing guo , Lei bai (2008)
6. A L Nielsen , K Steffensen, K L Largen , self assembling micro particles with controllable disruption properties based on cyclodextrin interaction.(2009)
7. Muzammil kuddushi, sargam Rajput ,ankit shah ,Jitendra p. mata AC sami(2009)
8. Bhattacharya , T.saha, P. Dash, J. Guanosine ,supramolecular hydrogel (2018)
9. L.Tan ,Y.Liu ,W .Ha, L.S.Ding , S.L. peng ,stimuli-induced gel -sol transition of multisensitive supramolecular β-cyclodextrin. soft matter(2012)
10. A. Harada, Y.Takashima ,H.Yamaguchi..(2009)
11. L. Jiang ,Y .Pang ,Y.Yan , M.Deng , Y .Wang .annular ring microtubes formed by β-cyclodextrin in aq.solution.(2010)
12. Lee,K.Y.Mooney, D.J. , hydrogel for tissue engineering .chem rev.(2001)
13. Li. J Kooger, R;he ,M;Xiao , X;zheng supramolecular hydrogel as a carrier deliver microRNA into cell. Chem (2014)
14. Tokarev, I gopyshetty , V Zhou, J Pita, M.Motornov, M;Kartz , E; Miniko ,stimuli responsive hydrogel membrain coupled with biocatalyst process ACS (2009)
15. Baral, A;bassak, S;Basu, K,Densorkhi,A;Hamely, I.W Banerjee, soft matter peptide based supramolecular gelator .soft matter (2015)
16. Li, J Geng ,L Wang ,G Chu , H Wei, self healable gel for use in wearable devises. chem matter.(2017)
17. Kopeek , J.Yang , J, peptide ,directed self assembly of hydrogel ,acta biomaterialla(2009)
18. Y.A.Gao, Z.H.Li, J.M Du , B.X. Han, G.Z.Li ,preparation and characterization of inclusion complex of β-cyclodextrin with ionic liquid.(2005)
19. Y.A.Gao, X.Zhao , B.dong ,L.Zheng, inclusion complexes of β-cyclodextrin with ionic liquid surfactant .J.Phy.chem.(2006)
20. M.Ao ,G.Xu, W.Kang, L.Meng, H .Gong ,surface rheological behavior of gelatin /ionic liquid type imidazolium gemini surfactant mixed system.soft matter (2011)
21. Dasgupta ,D Shrinivasan, S Rochas, C Ajayaghosh , A Guenet , hybrid thermoreversible gels from covalent polymers & organogels.langmuir (2009)
22. M kuddusi ,patel N.K, Rajput , S shah ,Malek N , thermo switchable de novo ionic liquid based gelator with dye absorbing and drug encapsulating characteristic, ACS omega (2018)
23. Li, L Yan , B Yang , J Huang ,W Chen .injectable self healing hydrogel with antimicrobial property.ACS Appl matter inter (2017)
24. Chen ,F Chen,Q Zhu ,L Tang ,Z Li ,Q Qin , G Yang .general strategy to fabricate strong and tough low molecular weight gelator based supramolecular hydrogel with double network structure .
25. Tuncaboylu , D C sari , M Oppermann, W Okay , tough and self healing hydrogel formed via hydroPhobic interaction macromolecules.(2011)
26. Valid Z , S Rajput , Muzammil kuddushi, M Kumar ,A El seoud ,Malek N I. synergistic interaction between cholesterol and functionalized ionic liquid based surfactant leading to the morPhological transition .chemistry select.(2018)
27. Adhikari , B Pauli, G Banerjee, self assembling tripeptide based hydrogel and their use in removal of dyes from waste water .soft matter.(2009)

28. Kumari ,H Klilne , S.R kennedy ,S R Garvey ,C Raston .manipulating three dimensional gel network entanglement by thin film shearing ,chem common (2016)
29. Gulyuz , U Okay ,O. self healing poly acrylic hydrogels.soft matter (2013)
30. Hongi Z , Hesheng X , Yue Z .poly(vinyl alcohol)hydrogel can automatically self heal. ACS macro left (2022)
31. Basak , S Nanda , J Banerjee A , multistimuli responsive self healing hydrogels tuning of the gel recovery property .chem common (2014)