CHAPTER 6

BIODEGRADATION STUDIES OF GUAR GUM BASED SUPERABSORBENTS USING SOIL BURIAL AND COMPOSTING METHODS
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6.1 Introduction

This chapter describes the biodegradation studies of semi-IPNs and IPNs prepared under different reaction conditions using soil burial and composting methods.

Hydrogels prepared from synthetic polymers only are not expected to be biodegradable. Degradation is a desirable requirement of hydrogels. Glycosidic linkages in the polysaccharide chain are degradable by microorganisms and hydrolytic enzymes. The degradation of polymers refers to any chemical change in polymer structure. Biodegradable polymers are defined as polymers that are degraded and catabolized, eventually to carbon dioxide and water, by microorganisms (bacteria, fungi) under natural environment and degraded product should not be harmful to the natural environment. Degradation may be caused by physical, chemical, mechanical and biological means. The presence of microorganisms is essential for biodegradation to take place and environment must provide the proper temperature, moisture level, oxygen (except for anaerobic bacteria) and nutrients. Biodegradation proceeds via hydrolysis and oxidation. Most biodegradable natural and biopolymers contain hydrolysable groups along the main chain. Linear polymers are generally more biodegradable than branched polymers. Albertsson and Karlsson (1997) have discussed the various factors affecting biodegradability.

Synthetic biodegradable PLA (polylactic acid), PGA (polyglycolic) and copolymers of these, have been manufactured for biomedical applications since the 1970s. Biodegradable polymers leave behind environment friendly by-products, such as CO₂, CH₄ and H₂O at the end of their life cycle (Mittal et al., 2013). Abouelwafa et al. (2008) studied the aerobic biodegradation of sludge from effluent produced during the processing of crude vegetable oil mixed with household waste obtained from landfill.
Different physico-chemical characteristics of the final composites were evaluated after 5 months of composting. Maiti et al. (2010) synthesized corn starch based biodegradable composites using Saccharum spontaneum L based graft copolymers and subjected them from biodegradation studies through composting method. They characterized different biodegradation stages through FTIR and SEM techniques. Synthesized natural fiber based composites were found to be biodegradable.

Fanta et al. (1971) has reported a comparative study based on biodegradation of starch, PEMA [poly(ethyl methacrylate)] and starch-g-ethyl methacrylate/sodium silicate (St-g-PEMA/SS). Biodegradation was carried out by three different methods: degradation in activated sludge, soil burial test and degradation by Bacillus cereus in culture medium. It was found that St-g-PEMA/SS showed accelerated rate of degradation (by weight loss), whereas, starch showed less amount of weight loss and a very negligible amount of weight loss was found in case of PEMA. Kale et al. (2007) studied the biodegradation of polylactic acid bottles under simulated conditions and compared the results with biodegradation under real composting conditions. They used cumulative measurement respirometric (CMR) and gravimetric measurement respirometric (GMR) simulated composting methods for the biodegradation studies. They observed that both methods exhibited similar trends of biodegradation and matches well with the theoretical degradation. Maharana and Singh (2006) have developed biodegradable polyethylene by graft copolymerization of starch and tested the biodegradability. Calandrelli et al. (2000) studied the preparation and characterization of composites based on biodegradable polymers for “in vivo” application. Biodegradable starch/clay based nano composite films were synthesized and used for food packaging applications (Avella et al., 2005). Bio based polymers are green and eco-friendly or biodegradable in nature. Biodegradable polymers have been used in biomedical applications due to their excellent biocompatibility, non-toxicity and low cost (Tian et al., 2012; Ulery et al., 2011). Biodegradation studies of the synthesized polymers were carried out using soil burial and composting methods keeping in view the importance of biodegradation studies of
polymeric materials. Characterization of the degraded samples at different stages of biodegradation was carried out using FT-IR and SEM techniques.

6.2 Results and Discussion

Synthesized crosslinked samples were subjected to biodegradation studies using soil burial and composting method. In case of soil burial method, soil was collected from one of the garden of NIT Jalandhar (Pb) and samples under investigation in the form of plates were buried in the soil. Soil was collected in the cleaned pots with an opening to the atmosphere as per the method mentioned under Chapter 3, Section 3.6.1. Tap water was supplemented every day to replenish the drying by evaporation throughout the period of 70 days. In case of composting method, compost was collected from the site of domestic waste-water effluent discharge at NIT Jalandhar (Pb). Samples were prepared in the form of plates and buried in the compost. The compost was collected in cleaned pots with an opening to the atmosphere as per the method mentioned under Chapter 3, Section 3.6.2. The microbial species in the compost were regularly fed with the aqueous solution from the effluent discharge as supplement to maintain the population balance and to keep the viability of culture. Biodegradation of the test samples was monitored both in soil burial and composting methods at different stages using FTIR and SEM techniques.

6.2.1 Biodegradation Studies using Soil Burial Method

Grafted samples like Ggum-cl-poly(AA)-AIR, Ggum-cl-poly(AA)-MW and Ggum-cl-poly(AA)-VO were found to degrade around 76.91%, 75.37% and 72.06% after the period of 70 days, respectively (Table 6.1, Figs. 6.1a, 6.2a and 6.3a). However, in case of grafting of poly(IA) chains onto Guar gum under different reaction conditions i.e., Ggum-cl-poly(IA)-AIR, Ggum-cl-poly(IA)-MW and Ggum-cl-poly(IA)-VO showed 74.55%, 70.44% and 70.23% degradation, respectively after 70 days (Table 6.1, Figs. 6.1b, 6.2b and 6.3b). Similarly, the IPNs prepared with PANI in neutral and
acidic medium by interpenetrating the polyaniline chains in between the different poly(vinyl) chains grafted on to Guar gum synthesized under different reaction conditions were found to degraded to the extent of 75-90% within the period of 70 days (Table 6.2, Figs. 6.1a-b, 6.2a-b and 6.3a-b).

Weights of test samples buried in burial soil were taken at a regular interval of 7 days and results are depicted in Tables 6.1-6.2. As expected, initially the weight of hydrogels was high due to moisture uptake. The networks start degrading later to such an extent that they could no longer hold a large amount of water and overall mass of the hydrogels got reduced. Degradation of polymers depends on several factors like pH, oxygen content, temperature, availability of mineral nutrients and humidity, which are responsible for the growth of microorganisms. The semi-IPNs and IPNs, synthesized under different reaction conditions degraded under soil burial method due to fragmentation of the large molecular species into small fragments of low molecular weights under the influence of bacterial digestion and cleavage of covalent bonds by active bacterial species present in the soil (Mittal et al., 2013a). Moreover, degradation of the test samples was found to depend upon the physical and chemical properties of the samples along with the percentage grafting of vinyl monomers onto backbone polymer and chemical reactivity of the monomeric species (Jeong et al., 1999; Kaith et al., 2010d).

6.2.2 Biodegradation Studies using Composting Method

Grafted samples like Ggum-cl-poly(AA)-AIR, Ggum-cl-poly(AA)-MW and Ggum-cl-poly(AA)-VO were found 81.64%, 83.07% and 82.67% degraded after the period of 70 days, respectively (Table 6.3, Figs. 6.1c, 6.2c and 6.3c). Ggum-cl-poly(IA) samples synthesized in air, under microwave and under vacuum was degraded to 80.06%, 82.94% and 78.47%, respectively after time period of 70 days (Table 6.3, Figs. 6.1d, 6.2d and 6.3d).
On the other hand, IPNs prepared in neutral and acidic conditions with polyaniline under different reaction conditions were found to degrade to the extent of 85-90\% after 70 days (Table 6.4, Figs. 6.1c-d, 6.2c-d and 6.3c-d). The maximum degradation (90.65\%) was observed in case of Ggum-cl-poly(AA-ipn-ANI)-AIR prepared in acidic medium. The degradation of semi-IPNs and IPNs synthesize under different reaction conditions due to the enzymatic and chemical decomposition of candidate samples. During soil burial and composting test, percentage weight loss calculation is an important method to check the biodegradation behaviour of materials. Moreover, biodegradation of test samples under microbe-rich composting media, test samples faced attack from the secretion products of micro-organisms, which resulted in cleavage of chemical bonds and bacterial degradation simultaneously.

6.2.3 Rate of Degradation using Soil Burial and Composting Methods

It has been observed that rate of degradation was in good agreement with percentage weight loss of semi-IPNs and IPNs. The percentage degradation of IPNs was higher as compared to semi-IPNs under soil burial method. Similarly, in case of IPNs, the rate of degradation was higher as compared to semi-IPNs under composting method. Rate of degradation was 1.003-1.098 mg/day in case of semi-IPNs under soil burial method (Table 6.1, Figs. 6.4a-b, 6.5a-b and 6.6a-b). Whereas, in case of IPNs, the rate of degradation was in the range of 1.125-1.263 mg/day (Table 6.2, Figs. 6.4a-b, 6.5a-b and 6.6a-b). On the other hand, the rate of degradation of IPNs was higher as compared to semi-IPNs under composting method (Table 6.3-6.4). The rate of degradation was in the range of 1.121-1.186 mg/day in case of semi-IPNs and 1.217-1.295 mg/day in case of IPNs (Table 6.3 and 6.4, Figs. 6.4c-d, 6.5c-d and 6.6c-d). Hence it has been observed that rate of degradation was faster in case of composting method in comparison to soil burial method.
Figs. 6.1a-d: Biodegradation Studies of Samples Synthesized in-Air (a-b) Soil Burial Method (c-d) Composting Method
Figs. 6.2a-d: Biodegradation Studies of Samples Synthesized under the Influence of Microwave Radiations (a-b) Soil Burial Method (c-d) Composting Method
Figs. 6.3a-d: Biodegradation Studies of Samples Synthesized under Vacuum (a-b) Soil Burial Method (c-d) Composting Method
Figs. 6.4a-d: Biodegradation Studies of Samples Synthesized in-Air (a-b) Soil Burial Method (c-d) Composting Method
Figs. 6.5a-d: Biodegradation Studies of Samples Synthesized under the Influence of Microwave Radiations (a-b) Soil Burial Method (c-d) Composting Method
Figs. 6.6a-d: Biodegradation Studies of Samples Synthesized under Vacuum (a-b) Soil Burial Method (c-d) Composting Method
<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Percentage Weight Loss at Different Time Intervals (Days)</th>
<th>Rate of Degradation per Day (mg)</th>
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<tr>
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<td>7</td>
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<tr>
<td>Ggum-cl-poly(AA)-AIR</td>
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<td>Ggum-cl-poly(AA)-MW</td>
<td>6.43</td>
<td>13.76</td>
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<td>Ggum-cl-poly(AA)-VO</td>
<td>6.96</td>
<td>13.09</td>
</tr>
<tr>
<td>Ggum-cl-poly(IA)-AIR</td>
<td>7.25</td>
<td>16.35</td>
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<tr>
<td>Ggum-cl-poly(IA)-MW</td>
<td>5.66</td>
<td>13.62</td>
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<td>Ggum-cl-poly(IA)-VO</td>
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Table 6.2: Biodegradation Studies of IPNs using Soil Burial Method

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<th>Sample Code</th>
<th>Percentage Weight Loss at Different Time Intervals (Days)</th>
<th>Rate of Degradation per Day (mg)</th>
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<tr>
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<td>14.04</td>
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<td>22.3</td>
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<td>Ggum-cl-poly(AA-ipn-ANI)-Neutral-VO</td>
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<td>16.36</td>
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Table 6.3: Biodegradation Studies of Semi-IPNs using Composting Soil Method

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<th>Sample Code</th>
<th>Percentage Weight Loss at Different Time Intervals (Days)</th>
<th>Rate of Degradation per Day (mg)</th>
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Table 6.4: Biodegradation Studies of IPNs using Composting Soil Method

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<td>8.63</td>
<td>16.15</td>
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<td>19.23</td>
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<td>Ggum-cl-poly(AA-ipn-ANI)-Acidic-VO</td>
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<td>18.03</td>
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1.217  
1.247  
1.241  
1.275  
1.25   
1.286
6.3 Evidences of Biodegradation

6.3.1 FT-IR Spectroscopy

6.3.1.a FTIR Studies of Semi-IPNs and IPNs Biodegraded under Soil Burial Method

FT-IR spectra of the samples at different stages of biodegradation were carried out in order to confirm the degradation of the cross-linked samples. It has been observed that the intensity of bands 3100-3600 cm\(^{-1}\) correspond to the –OH group of Guar gum and semi-IPNs kept on decreasing with increase in biodegradation period. The semi-IPNs and IPNs were degraded to the maximum extent in 50-70 days. Degradation of polymers depends on several factors e.g. pH, oxygen content, temperature, availability of mineral nutrients and humidity, which are responsible for the growth of microorganisms.

In case of Ggum-cl-poly(AA), the intensity of peaks observed at 3433 cm\(^{-1}\), 1547 cm\(^{-1}\), 1158 cm\(^{-1}\), 1723 cm\(^{-1}\), 1024 cm\(^{-1}\) and 811 cm\(^{-1}\) was found to shifted after biodegradation under different reaction conditions. In the final stage of biodegradation, most of the peaks observed initially were found missing or shifted and appearance of some new peaks were observed. This could be due to the reason that during biodegradation process, cleavage of bonds and hydrolysis take place simultaneously, which results in the formation of individual by-product components, hence shifting of peaks was observed (Kaith et al., 2010d). Similar behavior was observed in case of IPNs synthesized with PANI under different reaction conditions. The intensity of peaks at 1498 cm\(^{-1}\), 1725 cm\(^{-1}\) and 1156 cm\(^{-1}\) was found to shifted with degradation of the test samples. This was again due to the reason that cleavage of covalent bonds and hydrolysis take place simultaneously resulting in the formation of different type of by products (Kaith et al., 2010d). The transmittance of all the peaks was found to decrease and some peaks were shifted due to the breaking of the
covalent bonds and cross-linking under the influence of enzymes (Mittal et al., 2013a) (Figs. 6.7a-c, 6.9a-c and 6.11a-c).

In case of Ggum-cl-poly(IA), the intensity of peaks observed at 3429 cm\(^{-1}\), 2623 cm\(^{-1}\), 1705 cm\(^{-1}\) and 1309 cm\(^{-1}\) was found to be shifted. Most of the samples were found to get degraded to the maximum extent of about more than 75% after the 70 days period of degradation. At the final stage of biodegradation, most of the peaks observed initially were found missing or shifted and appearance of some new peaks was found due to breaking and disintegration of the semi-IPNs networks (Figs. 6.8a-c, 6.10a-c and 6.12a-c).

In case of samples where poly(IA) chains grafted onto Guar gum and their corresponding IPNs synthesized using interpenetration of PANI under different reaction conditions showed that intensity of peaks at 3432 cm\(^{-1}\), 1650 cm\(^{-1}\), 1498 cm\(^{-1}\) and 1544 cm\(^{-1}\) were found to be shifted and some of them are missing at the end of biodegradation period of 70 days due to breaking and disintegration of the crosslinked networks (Figs. 6.8a-c, 6.10a-c and 6.12a-c).

Hence, it is evident from FT-IR spectra that test samples got disintegrated under the hydrolytic and enzymatic conditions during biodegradation under soil burial method and was further supported through disappearance and shifting of peaks (Figs. 6.7a-c to Figs. 6.12a-c).
Figs. 6.7a-c: FTIR Spectra of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Burial Method
Figs. 6.8a-c: FTIR Spectra of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Burial Method
Figs. 6.9a-c: FTIR Spectra of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Burial Method
Figs. 6.10a-c: FTIR Spectra of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Burial Method
Figs. 6.11a-c: FTIR Spectra of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Burial Method
Figs. 6.12a-c: FTIR Spectra of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Burial Method
6.3.1.b FTIR Studies of Semi-IPNs and IPNs Biodegraded under Composting Method

In case of composting method, FTIR spectra of semi-IPNs and IPNs synthesized under different reaction conditions, at different biodegradation stages are depicted in Figs. 6.13a-c to Figs. 6.18a-c. Ggum-cl-poly(AA) exhibited characteristics peaks at 3433 cm\(^{-1}\), 1547 cm\(^{-1}\), 1158 cm\(^{-1}\), 1723 cm\(^{-1}\), 1024 cm\(^{-1}\) and 811 cm\(^{-1}\) and their corresponding IPNs synthesized through interpenetration of PANI exhibited characteristics peaks at 1498 cm\(^{-1}\), 1406 cm\(^{-1}\), 1725 cm\(^{-1}\), 1156 cm\(^{-1}\), 1027 cm\(^{-1}\) and 1158 cm\(^{-1}\). The change in the intensity of peaks after degradation stage was due to the disintegration of cross-linked network by the action of bacteria (Kaith et al., 2010). It has been observed from the comparison of FT-IR spectra of synthesized samples with biodegraded samples that variation in peaks intensity was due to the hydrolysis and enzymatic reactions during biodegradation and most of the peaks observed initially were found missing. Most of the bonds were easily accessible to the enzymes secreted during degradation by microbes (Maiti et al., 2010). It is quite evident from FT-IR studies that cross-linked samples got degraded to the maximum extent during the period of 70 days (Figs. 6.13a-c, 6.15a-c and 6.17a-c).

In the samples, poly(AA) chains grafted onto Guar gum and their corresponding IPNs using interpenetration of PANI synthesized under different reaction conditions exhibited characteristics peaks at 1498 cm\(^{-1}\), 2623 cm\(^{-1}\), 1705 cm\(^{-1}\), 1309 cm\(^{-1}\), 1025 cm\(^{-1}\), 1650 cm\(^{-1}\) and 1217 cm\(^{-1}\). These peaks were found to be shifted or missing at the final stage of degradation. This was due to the cleavage of some bond and formation of new bonds under the impact of enzymes existing in the bacterial rich compost culture (Figs. 6.14a-c, 6.16a-c and 6.18a-c) (Mittal et al., 2013b).

Therefore, it has been observed that cross-links and backbone started breaking down during the process of biodegradation the components of the test samples like vinyl monomeric chains, which was evident from weight loss. It has been further been supported from the FT-IR spectra of test samples during biodegradation process (Figs. 6.13a-c- Figs. 6.18a-c).
Figs. 6.13a-c: FTIR Spectra of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Composting Method
Figs. 6.14a-c: FTIR Spectra of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Composting Method
Figs. 6.15a-c: FTIR Spectra of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Composting Method
Figs. 6.16a-c: FTIR Spectra of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Composting Method
Figs. 6.17a-c: FTIR Spectra of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA), (b) Ggum-cl-poly(AA-ipn-ANI)-neutral and (c) Ggum-cl-poly(AA-ipn-ANI)-acidic using Soil Composting Method
Figs. 6.18a-c: FTIR Spectra of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA), (b) Ggum-cl-poly(IA-ipn-ANI)-neutral and (c) Ggum-cl-poly(IA-ipn-ANI)-acidic using Soil Composting Method
6.3.2 SEM Analysis

6.3.2.a SEM Studies of Semi-IPNs and IPNs under Soil Burial Method

Morphological changes in the semi-IPNs and IPNs synthesized under different reaction conditions were observed after biodegradation and are depicted in Figs. 6.19a-f to Figs. 6.24a-f. The initial stage of semi-IPNs and IPNs clearly showed the cross-linking and interpenetration of polyaniline within the hydrogel matrices synthesized under different reaction conditions. Breaking down of the three dimensional crosslinked structure during the degradation of cross-linked networks can be observed from SEM images. SEM images clearly exhibited the differences between smooth homogeneous surfaces of the hydrogels synthesized under different reaction conditions. The surface roughness and porosity was found to increases and also the surface became heterogeneous after the biodegradation of the cross-linked structures. This could be due to the breakage of cross-linking and covalent bonding between the backbone, cross-linker, poly(vinyl) chains and PANI by the action of active bacterial species present in the soil. The maximum extent of degradation of semi-IPNs and IPNs was found to be in the time interval of 70 days due to the liberation of CO₂, H₂O and humus of the soil. This was quite evident from the SEM analysis (Figs. 6.19a-f to Figs. 6.24a-f) (Mittal et al., 2013a).
Figs. 6.19a-c: SEM Images of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.19d-f: SEM Images of AIR Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
Figs. 6.20a-c: SEM Images of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.20d-f: SEM Images of AIR Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
Figs. 6.21a-c: SEM Images of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.21d-f: SEM Images of MW Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
Figs. 6.22a-c: SEM Images of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.22d-f: SEM Images of MW Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
Figs. 6.23a-c: SEM Images of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.23d-f: SEM Images of VO Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
Figs. 6.24a-c: SEM Images of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Burial Method
Figs. 6.24d-f: SEM Images of VO Synthesized Crosslinked (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Burial Method
6.3.2.b SEM Studies of Semi-IPNs and IPNs under Soil Composting Method

In case of composting conditions, the samples synthesized under different reaction conditions through graft copolymerization of poly(AA) and poly(IA) chains onto *Guar gum* and their corresponding IPNs synthesized with the interpenetration of PANI chains into the matrices in neutral and acidic medium clearly showed the morphological changes (Figs. 6.25a-f to Figs. 6.30a-f). The initial stage of semi-IPNs and IPNs clearly showed the cross-linking and interpenetration of the polyaniline within the hydrogel matrices. The changes in morphology, increased surface roughness of the samples, increased porosity and deepening of cracks on the samples along with weight loss during biodegradation at the time intervals of 70 days can be confirmed from the SEM images (Figs. 6.25a-f to Figs. 6.30a-f). These observed morphological changes were due to the reason already mentioned under Section 6.3.2.a (Mittal *et al.*., 2013a).

6.4 Comparative Studies of Biodegradation under Soil Burial and Composting Methods

On comparing the percentage degradation of semi-IPNs and IPNs in case of soil burial method, it has been observed that percentage degradation was higher in IPNs as compared to semi-IPNs synthesized under different reaction conditions. After the period of 70 days, both semi-IPNs and IPNs exhibited 70-77% and 75-90% degradation, respectively. IPNs showed higher degradation as compared to semi-IPNs due to the reason that PANI itself is not stable towards moistened conditions and easily degraded under bacteria attack. Also PANI interpenetrates into the semi-IPNs network, which makes it slightly more vulnerable to bacteria attack with the breaking of existing bond and formation of new bonds through secondary bonding forces. Hence more weight loss was observed through breaking of primary and secondary bonding. In case of semi-IPNs, there is maximum cross-linking between backbone
and poly(vinyl) chains. Hence the penetration of bacteria for biodegradation of the samples was not so easy. Therefore, the weight loss was more in case of IPNs as compared to semi-IPNs due to the presence of strong covalent bonding. This is evident from FTIR and SEM studies of these samples. Similar behavior of percentage degradation to be found in case of composting conditions i.e., the percentage degradation is more in case of IPNs as compared to semi-IPNs.

The maximum degradation has been found in case of Ggum-cl-poly(AA-ipn-ANI) prepared in acidic medium synthesized under different reaction conditions in comparison to other samples. This was due to the fact that poly(AA) chains are more hydrophilic in nature than poly(IA) and swells more in moistened conditions and thus leads to more microbial growth and activity, which resulting in maximum weight loss as compared to other samples.

It has also been observed that degradation of semi-IPNs and IPNs was higher in case of composting method as compared to soil burial method. It has been observed that rate of degradation was higher in case of composting method as compared to soil burial method (Tables 6.1-6.4). Presence of the large number of bacterial colony in compost degraded the samples through the cleavage of both primary and secondary bonding at a faster rate. This was supported from FTIR and SEM studies of the degraded samples (Figs. 6.7a-c to 6.30a-f).
Figs. 6.25a-c: SEM Images of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.25d-f: SEM Images of AIR Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method
Figs. 6.26a-c: SEM Images of AIR Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.26d-f: SEM Images of AIR Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method.
Figs. 6.27a-c: SEM Images of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.27d-f: SEM Images of MW Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method
Figs. 6.28a-c: SEM Images of MW Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.28d-f: SEM Images of MW Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method.
Figs. 6.29a-c: SEM Images of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(AA) Initial Biodegradation Stage; (b) Ggum-cl-poly(AA) Final Biodegradation Stage and (c) Ggum-cl-poly(AA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.29d-f: SEM Images of VO Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(AA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(AA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(AA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method
Figs. 6.30a-c: SEM Images of VO Synthesized Crosslinked Hydrogels (a) Ggum-cl-poly(IA) Initial Biodegradation Stage; (b) Ggum-cl-poly(IA) Final Biodegradation Stage and (c) Ggum-cl-poly(IA-ipn-ANI)-neutral, Initial Biodegradation Stage using Soil Composting Method
Figs. 6.30d-f: SEM Images of VO Synthesized Crosslinked Hydrogels (d) Ggum-cl-poly(IA-ipn-ANI)-neutral, Final Biodegradation Stage; (e) Ggum-cl-poly(IA-ipn-ANI)-acidic, Initial Biodegradation Stage and (f) Ggum-cl-poly(IA-ipn-ANI)-acidic, Final Biodegradation Stage using Soil Composting Method