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Synthesis of glycidyl triazolyl polymers using click chemistry

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Abstract—The glycidyl azide polymers converted easily to glycidyl 1,2,3-triazolyl polymers by the click chemistry in good to high yields. These reactions are affected deeply by the electron effects. The electron donating groups made the reaction faster.

1,2,3-Triazole compounds are important materials used widely as pharmaceuticals such as adrenergic receptor agonist,[1] antivirals,[2] antibacterials,[3] anti-HIV,[4,5] anticonvulsants,[6] and agrochemicals.[7] Moreover, compounds having 1,2,3-triazole group have found industrial applications as dyes, corrosion inhibitors, and photostabilizers.[8]

Polyethers containing pendant azide groups are cured by cycloaddition with diacetylenes to form triazole cross-linked binders.[9–11] These cross-linked polyethers have better mechanical properties and stability as well as higher burn rate than those of glycidyl azide polymers. The triazoles have high heat of formation (1*H*-1,2,3-triazole: +272 kJ/mol)[12] and triazole-based salts are used as energetic materials.[13,14] Glycidyl triazolyl polymers are expected to be good candidates for high energetic materials.

Recently, Sharpless group disclosed that attractive method for synthesis of the triazole ring.[15–18] This click reaction has very mild reaction conditions and is insensitive to water and oxygen. Moreover, this reaction gives easy access to regiospecific 1,4-disubstituted 1,2,3-triazoles. The synthetic method of the hyperbranched polymer having triazolyl units by polymerization with α,ω-diazidoalkane and tripropargylamine has been reported.[19] The direct synthetic method from the azide polymer bearing many azide groups by click reaction has been reported recently. However, to the best of our knowledge, the transformation of glycidyl azide polymer with phenyl acetylenes to glycidyl triazolyl polymer is still rare. Moreover, the electronic effects of the substituted groups on phenyl ring are not known clearly when coupling reaction takes place in polymer. So, we carried out to synthesize glycidyl triazolyl polymers from glycidyl azide polymers (GAPs) under click reaction conditions as shown in Scheme 1. The reaction of GAP (5 mmol for repeating unit, 1 equiv) with phenylacetylene (6 mmol, 1.2 equiv) in presence of sodium ascrobate (0.75 mmol, 0.15 equiv) and copper(II) sulfate pentahydrate (0.25 mmol, 0.05 equiv) in dichloromethane/water (1:1) at room temperature for 8 h gave the glycidyl-4-phenyl-1,2,3-triazolyl polymer[20] in 95% yield. The expected glycidyl triazolyl polymer could be obtained in high yields and all of azide groups in polymer converted to triazolyl groups clearly. This is the first case of the synthesis of glycidyl triazolyl polymer by click chemistry.

Polyepichlorohydrin (PECH) was used as the precursor for making GAP. PECH was prepared by ring opening polymerization of epichlorohydrin (ECH) using ethylene glycol (EG) as an initiator. The ratio of EG/ECH was 1:30. To introduce polymerization through the activated monomer mechanism,[21] ECH was added very slowly for 24 h by syringe pump. The number-average molecular weight (M_n) of the resulting product was 1231.

The GAP was synthesized by the azidation reaction of PECH with sodium azide in DMSO at 100 °C for 7 h. The completion of the reaction was confirmed by the disappearance of C–Cl peak (750 cm^{-1}) in IR spectra. The number-average molecular weight (M_n) of the resulting product was 1344.

Keywords: Glycidyl triazolyl polymer; Glycidyl azide polymer; Click chemistry.
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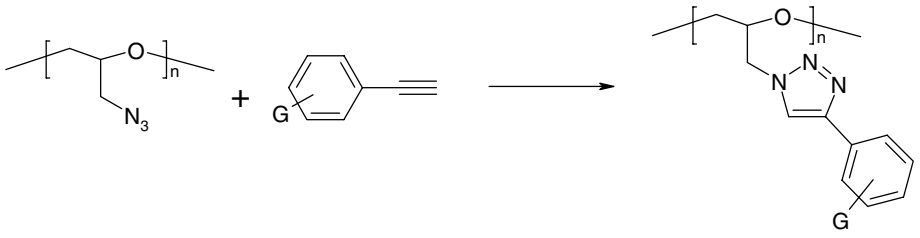
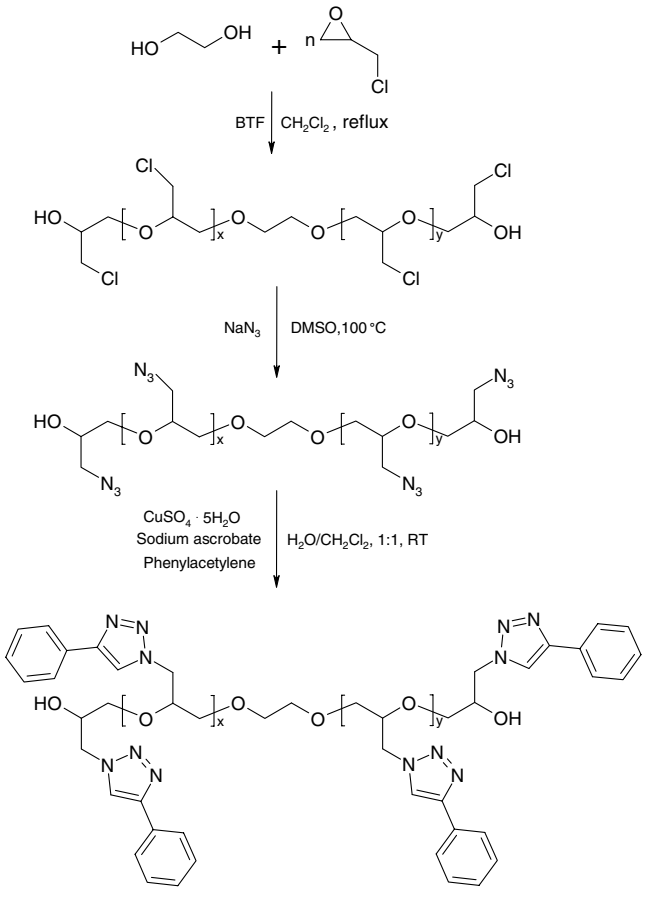


Scheme 1.

The copper-catalyzed azide–alkyne coupling is known to be efficient and easy to synthesize 1,2,3-triazole with various azide and alkyne species.[15–18] We used GAP containing azide functional groups in the main chain and 6 alkynes to obtain glycidyl-1,2,3-triazolyl polymer derivatives.

In general, the mixture of water and various organic solvents such as THF, CH_3CN, *tert*-butanol, ethanol, and DMSO have been used in click reaction as solvents. We, however, used the mixture of water and dichloromethane as the solvent system for this click reaction because of exceptional reactivity and facility of work up.[22]

First of all, phenylacetylene was applied to this reaction as shown in Scheme 2. The degree of the reaction progression was checked by IR spectrum. The samples were collected to get IR spectrum at time to time. The peak of azide (2100 cm^{-1}) disappeared clearly at 8 h after the reaction started as shown in Figure 3. So it took 8 h for the completion of the synthesis of glycidyl-4-phenyl-1,2,3-triazolyl polymer. The results of these click reactions are listed in Table 1.



Scheme 2.

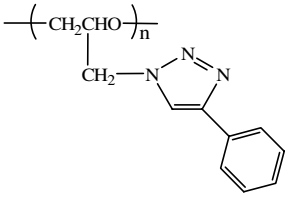
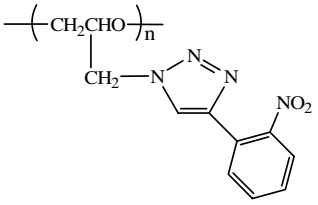
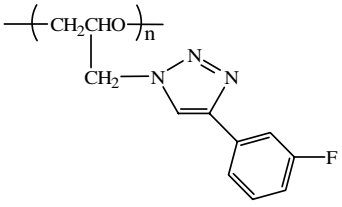
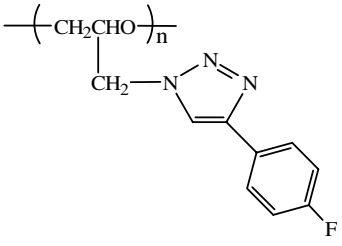
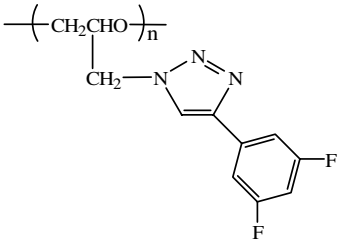
To know the reactivity, phenyl alkynes having electron-withdrawing groups in the phenyl group were applied to this coupling reaction. In the case of 1-ethynyl-2-nitrobenzene, even though the reaction went for 29 h, the azide peak in IR spectra did not clear and the trace of the peak remained. The alkynes having fluoro groups in the phenyl group, such as 1-ethynyl-3-fluorobenzene, 1-ethynyl-4-fluorobenzene, and 1-ethynyl-2,4-difluorobenzene worked slowly. Both of 1-ethynyl-3-fluorobenzene and 1-ethynyl-2,4-difluorobenzene took 27 h for the completion of the reaction. But, in the case of 1-ethynyl-4-fluorobenzene, the trace amount of the azide peak remained until 43 h. The completion time for phenyl acetylenes having electron-withdrawing groups was 4–5 times more than phenylacetylene. These results imply that these coupling reactions are deeply affected by the electron-withdrawing groups in the phenyl ring.

To know the electron-donating effect, 4-ethynyl-toluene and 1-ethynyl-4-methoxy-2-methylbenzene are also used in this reaction. The reaction completed after 6 h and 5 h, respectively. These reactions are faster than that of phenylacetylene. Even though we did not carry out for many electron-donating groups, we knew that the electron-donating groups make the reaction rate faster.

Other alkynes such as propagyl alcohol and 3-ethynyl aniline were also applied to these click reaction. Unfortunately, we could not obtain the completion of the reaction, because of the poor solubility of the products in water.

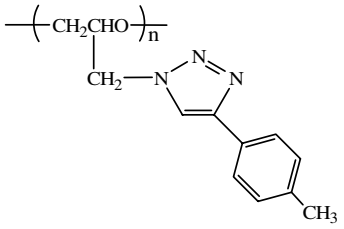
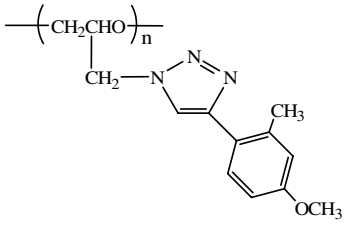
In general, the proton signals of 5-position of 1,2,3-triazoles appear 7.8–8.1 ppm.[23] However, unfortunately, in the case of glycidyl triazolyl polymers, we could not find their signals in proton NMR spectra because these signals located in the same region of the proton signals of phenyl rings as shown in Figure 1. In carbon NMR spectra, fortunately we could find clearly signals of 4- and 5-position of 1,2,3-triazoles. Their signals appeared at 140–148 ppm for 4-position and 115–125 ppm for 5-position with NOE effect as shown in Figure 2. The data showed that the structure of triazole in glycidyl triazolyl polymer is 1,4-substituted isomer. When changed to triazole group, the carbon signals of CH_2N_3 of GAP moved to 0.5 ppm upfield and appeared at 51.0 ppm.

Table 1. The results of the click coupling reaction from GAP with phenylacetylenes[a]

Run	Acetylene	Product	Reaction time (h)	M_n[b]	Yield[c] (%)
1	Phenylacetylene	 glycidyl-4-phenyl-1,2,3-triazolyl polymer	8	1569	95
2	1-Ethynyl-2-nitrobenzene	 glycidyl-4-(2′-nitrophenyl)-1,2,3-triazolyl polymer	29	1798	96
3	1-Ethynyl-3-fluorobenzene	 glycidyl-4-(3′-fluorophenyl)-1,2,3-triazolyl polymer	27.5	1865	95
4	1-Ethynyl-4-fluorobenzene	 glycidyl-4-(4′-fluorophenyl)-1,2,3-triazolyl polymer	43	1815	86
5	1-Ethynyl-3,5-difluorobenzene	 glycidyl-4-(3′,5′-difluorophenyl)-1,2,3-triazolyl polymer	26	2001	98

(*continued on next page*)

Table 1 (*continued*)

Run	Acetylene	Product	Reaction time (h)	M_n[b]	Yield[c] (%)
6	1-Ethynyl-4-toluene	 glycidyl-4-(4′-tolyl)-1,2,3-triazolyl polymer	6	1688	93
7	1-Ethynyl-4-methoxy-2-methylbenzene	 glycidyl-4-(4′-methoxy-2′-methylbenzyl)-1,2,3- triazolyl polymer	5	1951	95

[a] GAP/acetylene/$CuSO_4 \cdot 5H_2O$/sodium ascrobate = 1:1.1:0.05:0.15, 5 mmol scale, CH_2Cl_2/H_2O = 1:1, 50 ml.
[b] The values of M_n are measured based on polypropylene glycol standards.
[c] Isolated yields.

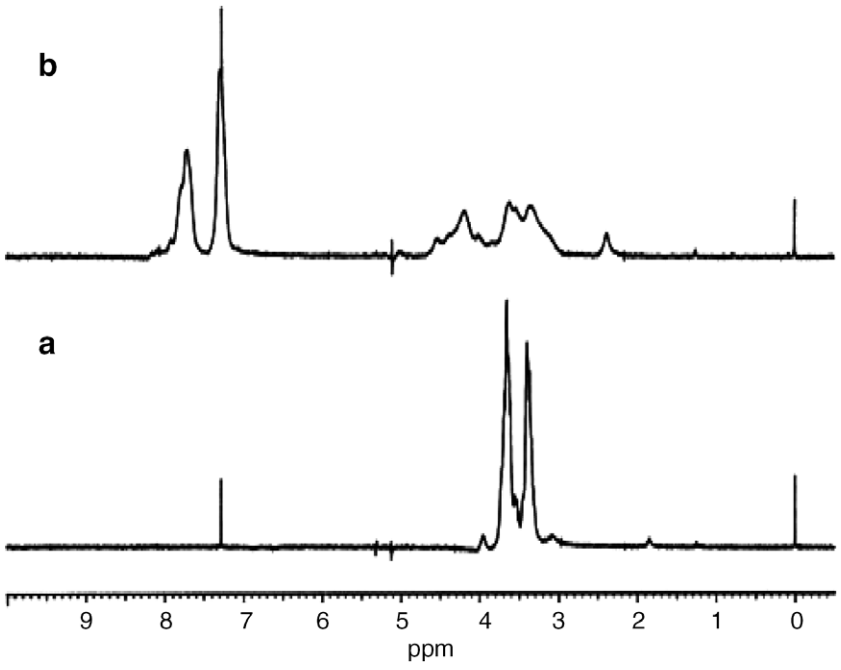


Figure 1. ^1H NMR in $CDCl_3$ of: (a) GAP; (b) glycidyl-4-phenyl-1,2,3-triazolyl polymer.

This signal movement is another evidence for the completion of the reaction.

In GPC trace, the number-average molecular weights of the products are revealed in the range of 1569–2001. As the example, the GPC trace of glycidyl-4-phenyl-1,2,3-triazolyl polymer (M_n = 1569) is shown in Figure 4. These results implied that the GAP changed to the triazolyl polymer by the coupling of alkynes.

Finally, another type of GAP was applied to these click coupling reactions as shown in Scheme 3. The GAP has functional groups such as acetyl group and azide group in the ends of the polymer. So it has no

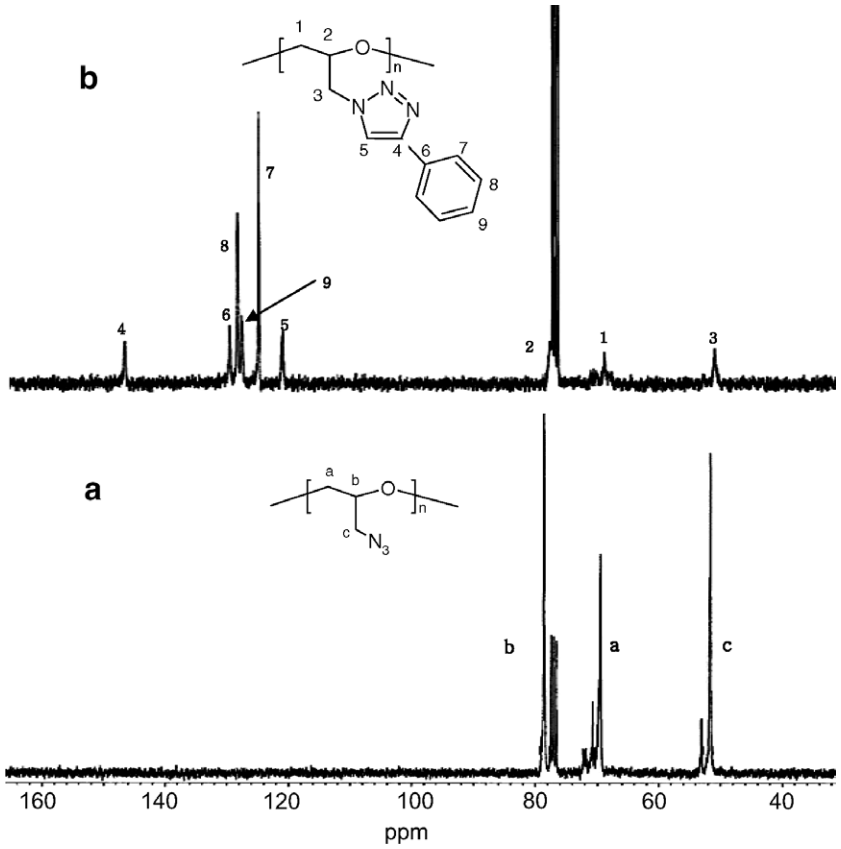


Figure 2. ^{13}C NMR in $CDCl_3$ of: (a) GAP; (b) glycidyl-4-phenyl-1,2,3-triazolyl polymer.

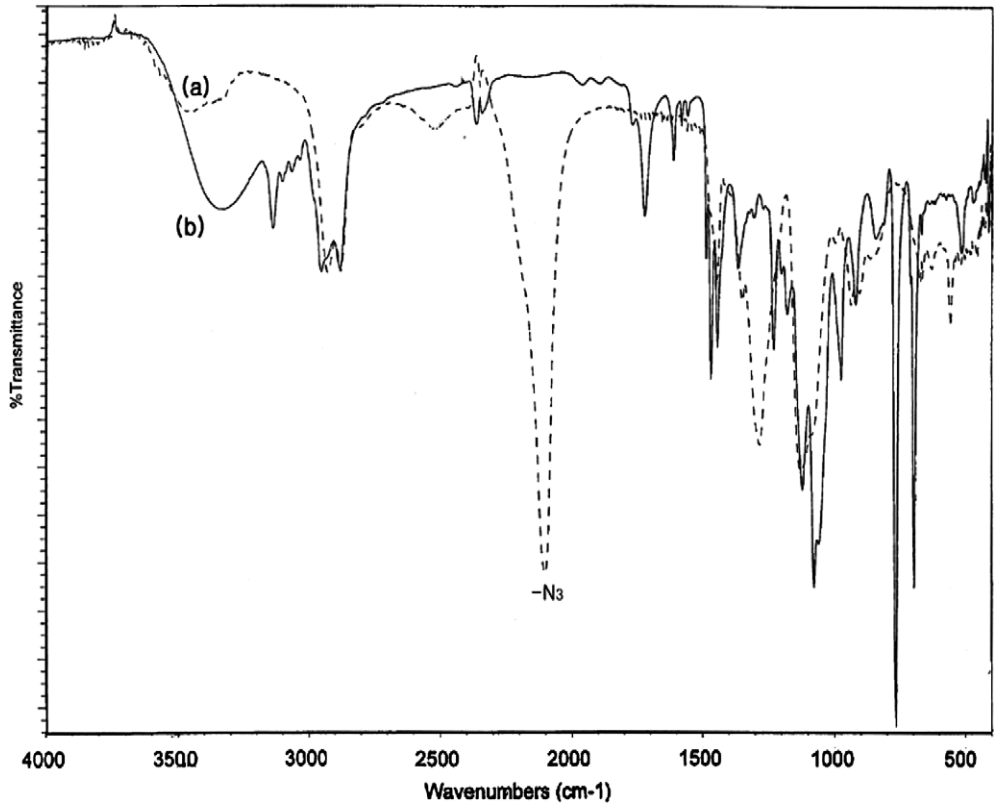


Figure 3. FT-IR spectra of: (a) GAP (dash line); (b) glycidyl-4-phenyl-1,2,3-triazolyl polymer (solid line).

terminal hydroxyl groups unlike above used GAP. This GAP also worked well with phenylacetylene to give the corresponding triazolyl polymer in 72% isolated yield.

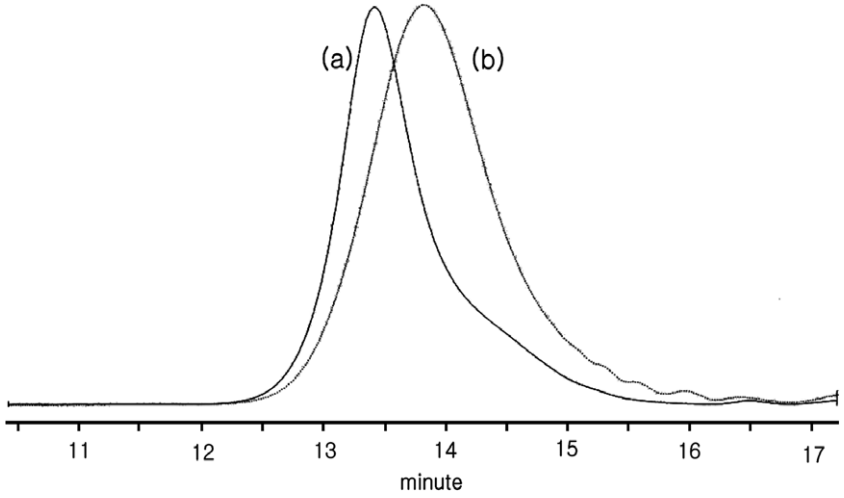
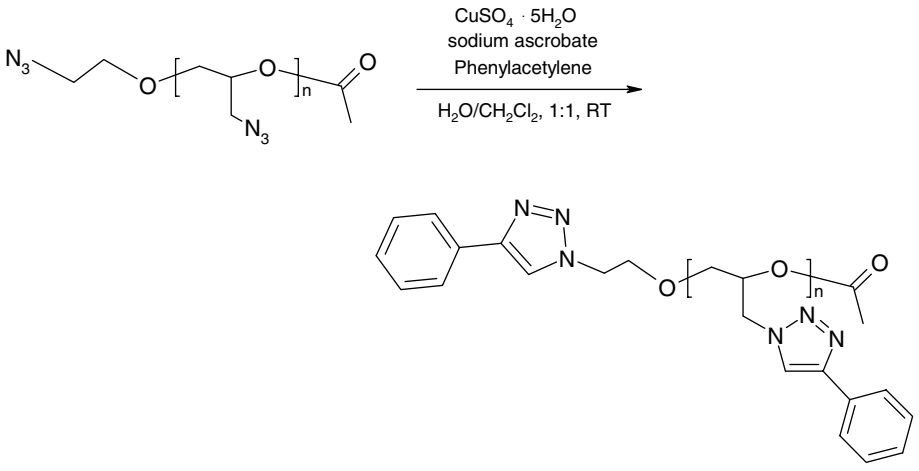


Figure 4. GPC traces of: (a) glycidyl-4-phenyl-1,2,3-triazolyl polymer ($M_n = 1569$); (b) GAP ($M_n = 1344$).



Scheme 3.

In conclusion, we found that the GAPs converted easily to glycidyl 1,2,3-triazolyl polymers by the click chemistry. The glycidyl 1,2,3-triazolyl polymers were obtained from various functional phenylacetylenes in good to high yields. These reactions are affected deeply by the electron effects. The substrates containing electron-withdrawing groups in the phenyl ring of phenylacetylene reacted with azide groups slowly. But, the electron donating groups made the reaction faster.

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20. *General procedure for click reaction.* GAP (0.5 g, 5 mmol for repeating unit, 1 equiv) was dissolved in dichloromethane (25 ml). Sodium ascrobate (0.15 g, 0.75 mmol, 0.15 equiv) and copper(II) sulfate pentahydrate (0.06 g, 0.25 mmol, 0.05 equiv) was dissolved in water (25 ml) and added into GAP solution. Finally, phenylacetylene (0.61 g, 6 mmol, 1.2 equiv) was added into the reaction mixture. The suspended mixture was stirred at room temperature for 8 h. The reaction mixture was poured into dichloromethane (10 ml), the organic layer was separated and washed with water (30 ml) in three times. The organic layer was concentrated and the concentrated solution was added into pentane (50 ml) in drops. The precipitate was collected by filtration and dried under vacuum to obtain the product as a pale-yellow powder (0.95 g, 95%). *Selected analytical data. Glycidyl-4-phenyl-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.20–7.40 (Hs of Ar and triazole), 7.25 (Hs of Ar), 5.00–2.90 (Hs of CH_2 and CH). ^{13}C NMR (75 MHz, $CDCl_3$) 147.27 (4-C of triazole), 130.17 (1-C of ph), 128.89 (3-C of ph), 128.20 (4-C of ph), 125.40 (2-C of ph), 121.45 (5-C of triazole), 77.81 (*C*H–O), 69.08 (*C*H_2), 51.01 (*C*H_2N). IR (KBr, cm^{-1}): 3331 (br, –OH), 3135 (w), 3095 (w), 3060 (w), 3029 (w), 2950 (m), 2879 (m), 1720 (w), 1610 (w), 1577 (w), 1549 (w), 1484 (m), 1465 (m), 1363 (w), 1227 (m), 1120 (s, C–O–C), 1076 (s), 974 (m), 919 (w), 844 (w), 766 (vs), 695 (s), 516 (w). $M_n = 1569$, PDI = 1.14. *Glycidyl-4-(2′-nitrophenyl)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 7.90 (s, 5-H of triazole), 7.74 (s, 3-H of Ar), 7.64 (s, 4-H of Ar), 7.48 (s, 5-H of Ar), 7.34 (s, 6-H of Ar), 4.80–3.10 (Hs of CH_2 and CH). ^{13}C NMR (75 MHz, $CDCl_3$) 148.01, 141.86, 135.41, 132.35, 130.72, 128.77, 124.31, 123.81, 77.50 (*C*H–O), 68.74 (*C*H_2), 51.18 (*C*H_2N). IR (KBr, cm^{-1}): 3402 (br, –OH), 3283 (w), 3142 (w), 2916 (m), 2878 (m), 1708 (s), 1616 (w), 1522 (vs, NO_2), 1457 (m), 1354 (s, NO_2), 1303 (w), 1225 (m), 1098, 1069 (s, C–O–C), 974 (m), 853 (s), 782 (s), 750 (s), 723 (m), 701 (m), 667 (m), 580 (w), 530 (m). $M_n = 1798$, PDI = 1.11. *Glycidyl-4-(3′-fluorophenyl)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.20–7.80 (4-H of triazole), 7.41 (s, 5, 6 Hs of Ar), 7.20 (s, 4-H of Ar), 6.87 (s, 2-H of Ar), 5.20–3.00 (Hs of CH_2 and CH). ^{13}C NMR (75 MHz, $CDCl_3$) 164.42, 161.27 (3-C of ph), 136.21 (4-C of triazole), 132.22 (1-C of ph), 130.45 (5-C of ph), 121.97 (6-C of ph), 121.01 (2-C of ph), 114.82 (5-C of triazole), 112.30, 112.00 (4-C of ph), 77.80 (*C*H–O), 68.92 (*C*H_2), 50.86 (*C*H_2N). IR (KBr, cm^{-1}): 3301 (br, –OH), 3138 (w), 2925 (m), 1620 (s), 1589 (s), 1485 (s), 1466 (m), 1447 (m), 1361 (w), 1307 (w), 1260 (m), 1230 (s), 1186 (w),1152, 1131, 1082 (s, C–O–C), 996 (w), 866 (vs), 786 (s), 737 (w), 686 (m), 527 (w). $M_n = 1865$, PDI = 1.11. *Glycidyl-4-(4′-fluorophenyl)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.20–7.50 (5-H of triazole and 2-Hs of Ar), 6.96 (s, 3-Hs of Ar), 5.20–3.00 (Hs of CH_2 and CH). ^{13}C NMR (75 MHz, $CDCl_3$) 164.33, 161.06 (4-C of ph), 166.77 (4-C of triazole), 127.40 (2-Cs of ph), 126.59 (5-C of triazole), 121.66 (1-C of ph), 116.16 (3-Cs of ph), 78.15 (*C*H–O), 69.34 (*C*H_2), 51.22 (*C*H_2N). IR (KBr, cm^{-1}): 3406 (br, –OH), 3138 (w), 3072 (w), 2921 (m), 1710 (s), 1611 (m), 1560 (s), 1497 (vs), 1459 (s), 1361 (s), 1296 (w), 1224 (s), 1157, 1124, 1097, 1076 (s, C–O–C), 974 (m), 841 (s), 815 (s), 765 (w), 604 (m), 528 (m). $M_n = 1815$, PDI = 1.11. *Glycidyl-4-(2′,4′-difluorophenyl)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.08 (4-H of triazole), 7.59 (s, 5, 6 Hs of Ar), 7.20 (s, 4-H of Ar), 6.87 (s, 2-H of Ar), 5.20–3.00 (Hs of CH_2 and CH). ^{13}C NMR (75 MHz, $CDCl_3$) 164.42, 161.27 (3-C of ph), 136.21 (4-C of triazole), 132.22 (1-C of ph), 130.45 (5-C of ph), 121.97 (6-C of ph), 121.01 (2-C of ph), 114.82 (5-C of triazole), 112.30, 112.00 (4-C of ph), 77.80 (*C*H–O), 68.92 (*C*H_2), 50.86 (*C*H_2N). IR (KBr, cm^{-1}): 3301 (br, –OH), 3138 (w), 2925 (m), 1620 (s), 1589 (s), 1485 (s), 1466 (m), 1447 (m), 1361 (w), 1307 (w), 1260 (m), 1230 (s), 1186 (w), 1152, 1131, 1082 (s, C–O–C), 996 (w), 866 (vs), 786 (s), 737 (w), 686 (m), 527 (w). $M_n = 2001$, PDI = 1.11. *Glycidyl-4-(4′-tolyl)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.20–7.40 (Hs of Ar and triazole), 7.09 (s, Hs of Ar), 5.05–2.90 (Hs of CH_2 and CH), 2.26 (br s, CH_3 of tolyl). ^{13}C NMR (75 MHz, $CDCl_3$) 147.32 (4-C of triazole), 138.00 (4-C of tolyl), 129.53 (3-C of tolyl), 127.27 (1-C of tolyl), 125.29 (2-C of tolyl), 121.07 (5-C of triazole), 77.81 (*C*H–O), 69.06 (*C*H_2), 51.03 (*C*H_2N), 21.30 (CH_3 of tolyl). IR (KBr, cm^{-1}): 3274 (br, –OH), 3138 (w), 3025 (w), 2920 (m), 2872 (m), 2244 (w), 1907 (w), 1499 (s), 1458 (s), 1411 (w), 1357 (m), 1318 (w), 1225 (s), 1120 (s, C–O–C), 1075 (s), 1046 (s), 974 (m), 910 (m), 821 (s), 799 (s), 732 (vs), 646 (w), 516 (m). $M_n = 1688$, PDI = 1.12. *Glycidyl-4-(4′-methoxy-2′-methylbenzene)-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 7.90–7.40 (Hs of Ar and triazole), 6.67 (Hs of Ar), 4.70–3.00 (Hs of CH_2 and CH), 3.76–3.70 (–OCH_3), 2.30 (br s, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) 159.07 (4-C of ph), 146.43 (4-C of triazole), 136.74 (2-C of ph), 129.69 (6-C of ph), 122.79 (1-C of ph), 122.08 (5-C of triazole), 116.11 (3-C of ph), 111.33 (5-C of ph), 77.85 (*C*H–O), 69.10 (*C*H_2), 55.11 (O*C*H_3 of ph), 50.86 (*C*H_2N), 21.57 (*C*H_3 of ph). IR (KBr, cm^{-1}): 3270 (br, –OH), 3138 (w), 3048 (w), 2998 (w), 2956 (m), 2836 (m), 1611 (s), 1574 (m), 1553 (m), 1493 (vs), 1456 (s), 1413 (w), 1350 (m), 1291 (s), 1241 (s), 1199 (w), 1167 (w), 1125 (s), 1073 (s), 1040 (s), 974 (m), 926 (w), 850 (m), 807 (m), 735 (m), 613 (w), 554 (w). $M_n = 1951$, PDI = 1.12. *Acetyl terminated Glycidyl-4-phenyl-1,2,3-triazolyl polymer*: ^{1}H NMR (300 MHz, $CDCl_3$) 8.00–7.55 (Hs of Ar and triazole), 7.31 (Hs of Ar), 5.02–3.00 (Hs of CH_2 and CH), 1.83 (CH_3 of acetyl). ^{13}C NMR (75 MHz, $CDCl_3$) 169.67 (*C*=O of acetyl), 147.42 (4-C of triazole), 130.22 (1-C of ph), 128.87 (3-C of ph), 128.18 (4-C of ph), 125.45 (2-C of ph), 121.47 (5-C of triazole), 77.94 (*C*H–O), 69.68 (*C*H_2), 50.97 (*C*H_2N), 20.81 (*C*H_3 of acetyl). IR (KBr, cm^{-1}): 3134 (br, –OH), 3099 (m), 3060 (w), 3029 (w), 2951 (w), 2912 (w), 2877 (m), 2248 (w), 1743 (m, C=O), 1483 (s), 1465 (s), 1441 (m), 1369 (w), 1228 (s), 1126, 1077, 1045 (s, C–O–C), 973 (m), 911 (m), 810 (s), 765 (vs), 732 (s), 695 (s), 512 (w). $M_n = 959$, PDI = 1.08.
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