

BUKU PROFIL PENYELIDIKAN SKIM GERAN PENYELIDIKAN GERAN UNIVERSITI JANGKA PENDEK / GERAN DALAM UMP

SYNTHESIS AND CHARACTERIZATION OF NI-BASED CATALYSTS MODIFIED WITH RARE EARTH AND ALKALINE METAL OXIDES (CeO₂, La₂O₃, BaO) FOR METHANE CRACKING

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ABSTRACT (120 words)

Nickel (Ni) has been recognized as a low-cost and an efficient catalyst for hydrogen production from methane cracking. Nevertheless, methane cracking reaction is still hindered by accumulation of carbon on Ni catalyst, which is produced during the reaction. Modification of Ni catalyst with other substance known as promoter is essential to reduce the carbon deposition. Additionally, supported catalyst with relatively large surface area and highly catalyst dispersion for example is essential to develop carbonresistance Ni-based catalysts. In order to evaluate the effect of promoter, Ni metal has been modified with cerium (Ce), lanthanum (La) and barium (Ba) and a self-combustion process, glycine- nitrate process (GNP) has been employed for the catalyst preparation method. Effects of glycine-nitrate (G/N) ratio and calcination temperature have been investigated on Ni catalyst properties and morphology. Ni catalyst with modified La supported on an inert support, SiO₂ (Ni-La/SiO₂) was further chosen for catalytic activity in methane cracking. In situ self-combustion has been employed where the inert SiO_2 support is immersed into the fuel-nitrate solution where the solution is allowed to selfignited. Effects of support sizes, support loading, La loading and reaction gas concentration have been evaluated on catalyst activity and carbon formation. All Ni catalysts modified with Ce, La and Ba are very high in crystallinity. The reduced catalyst of Ni catalyst modified with Ce composed of Ni and CeO₂, the reduced Ni modified with La catalyst composed of separated phases of Ni and La₂O₃ while the reduced Ni catalyst modified with Ba composed of Ni and BaN₂O₆. The Ni-Ba catalyst however does not contain BaO as expected. In all catalysts, morphology of the catalyst is highly influenced by the G/N ratio. Catalyst with G/N= 0.5 ratio displays a foam-like open-pore structure with non-uniform macropores while G/N= 1.5 produce catalyst with small pores with the presence of denser surfaces. G/N=1.0 ratio is the optimum G/N as it produced catalyst with large and more uniform macropores. Meanwhile 800°C was found to be the best calcination temperature for Ni metal catalysts modified with Ce, La and Ba. From catalytic activity through methane cracking, Ni-La/SiO₂ supported with SiO₂ support with a lower particle size has a better conversion than the shown by Ni-La/SiO₂ with a support of larger particle size. Initial H₂ yield for Ni-La/SiO₂ supported with SiO₂ support with a lower particle size was higher compared that for by Ni-La/SiO₂ with a support of larger particle size. Catalyst dispersion calculated for Ni-La/SiO₂ supported with SiO₂ support with a lower particle size is higher than the one with a support of larger particle size, suggesting that catalyst supported on a support of high surface area has a better catalyst dispersion. Higher support loading also gave better catalyst dispersion. Nevertheless, massive amount of filament carbons is found on the surface of Ni-La/SiO₂ supported with SiO₂ support with a lower particle size catalyst suggesting that the catalyst is very active for catalytic activity. In Ni/SiO2 without La, high initial methane conversion of 58% was achieved yet the conversion was drop gradually to 30% after 150 minutes of reaction time. As 5% of La was added into Ni catalyst, the methane conversion was maintained at lower ~40% throughout the experiment. Further increase of La to 20% has reduced the methane conversion to ~38%. Reaction gas composition CH_4 : N₂= 1:2 have the highest methane conversion initially at ~75% and as the methane concentration decreased, the initial methane conversion decreased. In conclusion, the work has successfully synthesized Ni modified with La catalyst supported on SiO₂ (Ni-La/SiO₂) using in situ glycine- nitrate process of high dispersion, thus resulted in a decent catalytic activity in methane cracking.

1. INTRODUCTION

Nickel-based catalyst is an excellent catalyst for hydrogen production due to its promising catalytic performance in terms of conversion and selectivity [1] and less expensive [2]. Although the catalyst is a favorable for hydrocarbon processing, Ni alone is very prone to carbon deposition. In order to take advantages of Ni good properties, the modification of Ni catalyst with other substance known as carbon inhibitor is essential to reduce the carbon deposition.

Ni catalyst highly dispersed with ceria (CeO₂) has shown to have a high resistance towards carbon deposition [3]. Ceria is able to shift easily between reduced and oxidized state (Ce³⁺ \Leftrightarrow Ce⁴⁺) and accommodate variable levels of bulk and surface oxygen vacancies thus improving catalytic performances [4]. Carbon formation was significantly reduced and the interaction of carbon and nickel is weaker with the presence of ceria [5]. Metallic catalysts promoted with cerium are interesting due to cerium properties influence the mechanic and thermal resistance of support, metallic dispersion, catalytic performance, and carbon reduction on the surface catalyst [6].

Addition of La_2O_3 to Ni/SiO₂ increased the dispersion of Ni on support and decreasing the assemblage of Ni particles during the methane cracking reaction [7]. Modifying a Ni/ α -Al₂O₃ catalyst by La_2O_3 has enhanced the Ni particles dispersion and led to strong metal support interaction [8]. Furthermore, addition of La increases spacing between active nickel particles and prevents agglomeration and sintering [8]. The addition of La_2O_3 has also increased the reactant conversion due to small NiO particles with strong metal support interaction enhanced dissociative adsorption of methane leading to relevant catalytic activity with appreciable methane conversion and an optimal H₂ yield.

The catalytic activity and stability of Ni catalysts were found to be greatly enhanced by the addition of barium [9]. The presence of Ba species in catalysts increased the conversion of CO, NOx and hydrocarbons and suggested that the addition of barium has enhanced the basicity of catalysts. In addition, that barium can help to suppress the sintering of the catalysts. The mobility of atoms could be reduced by barium, which suppresses the formation of larger particles at high-temperatures [10].

Self-combustion has become an attractive and very popular synthetic method for preparation of catalyst powder [11]. As one of the most popular self-combustion methods; glycine nitrate combustion process (GNP) uses amino glycine as a fuel and metal nitrates of the targeted composition as an oxidant for combustion process. Physical properties of the synthesized catalyst powders are directly influenced by the process constituents. The average particle size decreases and the specific surface area increases with increasing glycine amount in the starting composition. The glycine-to-nitrate (G/N) ratio is known to have an effect on the flame temperature, combustion rate and product morphology of the catalyst produced [12]. Calcination is a thermal treatment process which takes place at temperatures below the melting point to bring about thermal decomposition, phase transition or removal of volatile fractions of the catalyst. Calcination temperature significantly affects the texture property and the phase composition of catalysts [13].

Ni-supported catalysts produced using impregnation, co-precipitation and sol-gel have relatively low specific surface area [14] or poor dispersion of Ni-particles [15]. Under hydrocarbon atmosphere, low dispersed catalyst is more likely to form more carbon on catalyst surface [16]. Hence, a highly dispersed Ni-based supported catalyst is essential

to provide carbon-resistance catalysts, thus catalyst preparation method which produces as such catalyst morphology should be employed. In-situ glycine-nitrate combustion process is a recent catalyst synthesis process in which metal-supported catalyst is produced through a combination of impregnation and self-combustion approaches [17]. A rapid high temperature reaction results in metal particles deposited onto the support. Highly dispersed metal particles as a result of a chemical reaction propagates through the support due to rapidly moving combustion wave [18]. In-situ combustion synthesis is a cost effective method [19] which display magnificent properties with respect to crystallite size and degree of reduction and metal dispersion [20].

The objectives of the entire project are: (1) to synthesis and characterize Ni-based catalysts modified with La, Ce, and Ba using glycine-nitrate combustion process. (2) to synthesize and characterize Ni-supported catalyst modified with selected promoter using in situ glycine-nitrate combustion process (in situ GNP); (3) to investigate the catalytic performance and and carbon formation of Ni-supported catalyst modified with selected promoter in methane cracking. The focus of this work is to produce Ni supported catalyst with high dispersion thus stable towards methane cracking and carbon formation.

2. RESEARCH METHODOLOGY

The work begins with synthesis and characterization of Ni catalysts modified with promoters; La, Ce and Ba using glycine-nitrate combustion process (GNP). Support was yet to be included in the preparation. La-modified, Ce-modified and Ba-modified Ni catalyst were synthesized using GNP with solution precursor acts as the oxidizer and glycine as the fuel. The precursor is a solution containing lanthanum nitrate (La(NO₃)₃).6H₂O), cerium nitrate hexahydrate (Ce(NO₃)₃.6H₂O) or barium nitrate Ba(NO₃)₂ and nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O) (Ni-0.90, CeO₂-0.10) in a deionized water. Glycine (NH₂CH₂COOH) is added into the precursor and stirrer to produce a glycine-nitrate solution. The glycine nitrate solution was then heated using a hot plate at 90°C and stirred for overnight until a viscous gel of glycine-nitrate solution was formed. The gel was then transferred into a ceramic container and further heated at higher temperature (180°C) until the gel solution self-ignited. The combustion of the gel resulted in catalyst powder and large amount of released of gases. Glycine was then added into the homogeneous solution of nickel nitrates at various glycine-to-nitrate (G/N) ratios (G/N=0.5, G/N=1.0, G/N=1.5). Catalyst powders were calcined for 2 hrs at various calcination temperatures (600, 700, 800°C). The physical properties and morphology of the synthesized catalysts were characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM). Glycine-nitrate combustion process (GNP) of Ni catalyst is shown in Figure 2.1.



Figure 2.1 Glycine-nitrate combustion process (GNP) of Ni catalyst

Ni-supported catalyst modified with selected promoter were synthesized using in situ glycine-nitrate combustion process. Ni-La catalysts supported on SiO₂ were synthesized using nickel nitrate hexahydrate (Ni(NO₃)₂.6H₂O), glycine (C₂H₅NO₂), lanthanum nitrate hexahydrate, La(NO₃)₃ · 6H₂O and silica (SiO₂) as raw materials. Initially, nickel nitrate hexahydrate and glycine were dissolved in deionized water to form a reaction media solution. Porous solid silica (SiO₂) was added to the reaction media solution. The support-reaction media was stirred and heated overnight at 90°C until it forms a gel solution. The gel solution was then heated at 180°C until the solution was self-ignited and combusted to produce an ash catalyst powder. The ash powder was then calcined at 800°C. In this part, effects of support particle size, catalyst-to-support ratios and La loadings were varied. Two types of commercial SiO₂ support were used in this study; SiO₂ gel (particle size: 74 µm, designated as SiO₂ B). Synthesis of Ni-La/SiO₂ using in situ glycine-nitrate combustion process is given in Figure 2.2.



Figure 2.2 Synthesis of Ni-La/SiO₂ using in situ glycine-nitrate combustion process

Catalytic performance of Ni-La/SiO₂ was evaluated in methane cracking in a fixed-bed quartz tube reactor system. Catalyst was reduced under a mixture of 10% H_2/N_2 at 700 °C for 2 h before contacted to the reaction gas of 20% CH₄/N₂ at a total flow rate of 60 mL/min. Methane cracking was conducted at 500 °C for 6 h and the process was maintained at 45,000 mL/g·h of gas space hour velocity (GHSV). The product gas was collected throughout the experiment using gas bags and the composition of the product gas was determined by gas chromatograph (GC) equipped with thermal conductivity detector (TCD) (Agilent Technologies 6890 series, USA) using molecular sieve 13X column (Agilent, USA). Experimental setup of methane cracking for Ni-La/SiO₂ catalyst is shown in Figure 2.3.



Figure 2.3 Experimental setup of methane cracking for Ni-La/SiO₂ catalyst

3. FINDINGS

Figure 3.1 shows the XRD patterns for Ni catalysts modified La, Ce and Ba (Ni-La, Ni-Ce and Ni-Ba) synthesized using GNP. After the calcination process, La element in Ni catalysts modified with La became visible in a form of LaNiO₃. The peak of LaNiO₃ given by (100) plane at 33° became more obvious as the calcination temperature was increased from 600 to 800°C. After the reduction process, the peak representing LaNiO₃ was entirely diminished while the peak belongs to La₂O₃ at (220) plane was detected at 77°. Meanwhile for Ni-Ce, the CeO₂ phase in Ni modified catalyst was produced directly from the GNP. The CeO₂ remains after the reduction process and presence together with Ni in two separated phases (Ni and CeO₂) in the Ni modified Ce catalyst. For Ni-Ba catalyst, the peaks belong to BaCO₃ phase were detected at 24.2°, 34.2° and 46.8° calcined at 600 and 700°C. The peaks however were diminished when the calcination temperature was increased to 800°C. At the same time, new peaks belong to BaN₂O₆ phase were appeared in the catalyst calcined at 800°C. Therefore, barium-containing compound in Ni-Ba catalyst calcined at 800°C existed as BaN₂O₆ phase. In all catalysts, XRD patterns show that the peaks become higher and sharper as the calcinations

temperature increases suggesting the crystallinity of the catalyst has increased with the increased of calcination temperature.



Figure 3.1 XRD patterns for Ni catalysts modified La, Ce and Ba (Ni-La, Ni-Ce and Ni-Ba) synthesized using GNP

SEM micrographs of Ni catalysts modified with La, Ce and Ba at various glycine-nitrate (G/N) ratios are shown in Figure 3.2. Glycine-to-nitrate (G/N) ratio has a pronounce effect on the morphology of the catalyst. At G/N ratio of 0.5, both Ni-La and Ni-Ce catalysts have 'cottonwool-like' structures. As the G/N ratio increased from 0.5 to 1.0, the catalysts formed a 'spongy-like' structure with random-size pores. At G/N= 1.5, both catalysts become denser with much lesser pores on their surfaces. The 'cottonwool-like' and highly porous structure observed for the catalysts at G/N=0.5 is due to the low flame temperature, which is associated with fuel-lean composition. As the G/N ratio increases to G/N= 1.0, close to the stoichiometric ratio, the structure changed to 'spongy-like' structure as the agglomeration increases due to higher flame temperature. At G/N ratio above the stoichiometric ratio (G/N=1.5), the flame temperature decreases again with the increasing of fuel due to the incomplete combustion. At fuel-rich composition (G/N=1.5), the catalyst become dense and the porosity was the least. In Ni-Ba catalyst, all the catalysts exhibited highly porous microstructure, but with different morphology. Catalyst with G/N= 0.5 ratio has a foamy structure and spongy in nature with micropores. The G/N= 1.0 on the other hence produces catalyst with spongy surfaces and larger pores on the surface. The morphology for the catalyst with G/N= 1.5 was also similar with one produced using G/N= 1.0. Unlike Ni-La and Ni-Ce catalysts, Ni-Ba catalyst maintained a spongy structure yet with larger and more pores as the G/N decreases.



Figure 3.2 SEM images of Ni catalysts modified with La (a - c), Ce (d - f) and Ba (g - i) at glycine-nitrate ratio (G/N) = 0.5, 1.0 and 1.5.

XRD patterns for Ni–La catalyst supported on two supports of different particle size; SiO₂ B (Ni–La/SiO₂ B) and SiO2 A (Ni–La/SiO₂ A) are given in Figure 3.3. The Ni–La/SiO₂ A and Ni–La/SiO₂ B catalysts are synthesized using in situ glycine–nitrate combustion and composed of an amorphous SiO₂, Ni metallic, and La₂O₃ phases. The diffraction peaks for Ni phase in Ni–La/SiO₂ A and Ni–La/SiO₂ B became wider with the addition of supports. The introduction of SiO₂ support has reduced the crystallinity of Ni metal in Ni–La/SiO₂ catalysts, which also suggests that Ni metal catalyst has been highly dispersed over the surface of SiO₂ support in the Ni–La/SiO₂ catalysts.



Figure 3.3 XRD patterns for Ni–La catalyst supported on two supports of different particle size; SiO₂ B (Ni–La/SiO₂ B) and SiO₂ A (Ni–La/SiO₂ A)

The calculated catalyst dispersions for Ni–La/SiO₂ A and Ni–La/SiO₂ B at various catalyst-to-support ratios are shown in Figure 3.4. Catalyst dispersion calculated for Ni–La/SiO₂ supported with SiO₂ support with a lower particle size is higher than the one with a support of larger particle size, suggesting that catalyst supported on a support of high surface area has a better catalyst dispersion. Higher support loading also gave better catalyst dispersion.



Figure 3.4 Calculated catalyst dispersions for Ni–La/SiO₂ A and Ni–La/SiO₂ B at various catalystto-support ratios

Catalytic performances of the Ni–La/SiO₂ catalysts of different support size were evaluated in methane and methane conversion and H2 yield for Ni–La/SiO₂ A and Ni–La/SiO₂ B are shown in Figure 3.5. The methane conversions for Ni–La/SiO₂ A were within the range of 20%–40%. Ni–La/SiO₂ supported with SiO₂ support with a lower particle size (Ni–La/SiO₂ B) exhibited a higher methane conversion (maximum of ~60%) during the first 120 min before it decreased to ~30% and maintained until the end of the reaction. Support with high surface area provides a high number of catalytic active sites for metal-supported catalyst. A better hydrogen yield was produced by Ni–La/SiO₂ supported with SiO₂ support with a lower particle size (Ni–La/SiO₂ B) when the catalyst gave an approximately 10% of hydrogen yield at the beginning of the reaction before the value decreased and became constant at ~4%. Catalyst with high support surface area provided more active sites for the hydrogen production to continuously take place during the cracking reaction.



Figure 3.5 Methane and methane conversion and H2 yield for Ni–La/SiO₂ of different support size.

FESEM images of fresh and spent Ni–La/SiO₂ A and Ni–La/SiO₂ B catalyst is shown in Figure 3.6. In fresh catalyst, dense structure with a smooth surface belongs to the SiO₂ support while the porous-like structure on the surface of the support is the Ni–La catalyst. Support with a high surface area would provide a better distribution of catalyst particles with a higher chance of better catalyst dispersion. Spent Ni–La/SiO₂ A comprises of spherical-like structure due to the Ni particles and fibers identified as filament carbons. Spherical Ni particles available on the catalyst surface were the result of the Ni particles detachment from the bulk of Ni due to the activity of the filament carbons and explains the very low H₂ yield by Ni–La/SiO₂ A during the methane cracking. A massive amount of filament carbons was found on the surface of Ni–La/SiO₂ B suggesting that the catalyst had been active for both methane cracking and carbon formation. Nevertheless, the detachment of the Ni particles was not observed on the spent Ni–La/SiO₂ B, thus giving a better H₂ yield compared with that of Ni–La/SiO₂ A.



Figure 3.6 FESEM images of fresh and spent Ni–La/SiO₂ A and Ni–La/SiO₂ B catalysts

Figure 3.7 shows the effect of La addition on methane conversion and H₂ yield. Ni catalyst without promoter, Ni/SiO₂ was initially achieved the highest methane conversion which is 58%. However, the conversion was drop gradually to 30% after 150 minutes of reaction time. As 5% of La was added into Ni catalyst, the methane conversion was lower which is ~40% and maintains throughout the experiment. Further increase of La to 20% has reduced the methane conversion to ~38%. The highest hydrogen yield which is 18% was achieved over Ni catalyst without La addition. However, the H₂ yield was dropped drastically (to 0%) after 120 minutes of reaction time. With addition of 5% of La to the Ni catalyst, H₂ yield was decreased to 10%, however the stability of the catalyst was increased. Further increase of La to 20% deactivates the catalyst and reduces the lifetime of the catalyst to only 90 min reaction time. The presence of La decreases the

hydrogen yields to about 10% because some of Ni surface area has been covered by La.



Figure 3.6 Methane and methane conversion and H2 yield on effect of La loading.

Figure 3.7 shows the effect of reaction gas composition on methane conversion and H₂ yield of Ni-La/SiO₂. Reaction gas composition CH₄: N₂= 1:2 have the highest methane conversion initially at ~75% before drops to ~15% methane conversion. As the methane concentration decreases from high concentration (CH₄: N₂= 1:2) to lower concentration (CH₄: N₂= 1:4, 1:7) the initial methane conversion decreases. As the concentration of methane increases, the hydrogen yield decreases.



Figure 3.7 Methane and methane conversion and H2 yield on effect of reaction gas composition

4. CONCLUSION

In summary, Ni catalysts been modified with cerium (Ce), lanthanum (La) and barium (Ba) has been successfully synthesized using a self-combustion process, glycine- nitrate process (GNP). In all catalysts produced are high in crystallinity and crystallinity of the catalyst has increased with the increased of calcination temperature. 800°C was found to be the best calcination temperature for Ni metal catalysts modified with Ce, La and Ba. The Ni catalyst modified with Ce composed of Ni and CeO₂, the Ni modified with La catalyst composed of separated phases of Ni and La₂O₃ while the Ni catalyst modified with Ba composed of Ni and BaN₂O₆. Ni catalyst with modified La supported on an inert support, SiO₂ (Ni-La/SiO₂) was further chosen for catalytic activity in methane cracking. The catalyst was successfully produced using in situ glycine- nitrate process (in situ GNP). Ni-La/SiO₂ supported with SiO₂ support with a lower particle size has a better conversion than the shown by Ni-La/SiO₂ with a support of larger particle size. Initial H_2 yield for Ni-La/SiO₂ supported with SiO₂ support with a lower particle size was higher compared that for by Ni-La/SiO₂ with a support of larger particle size. Nevertheless, massive amount of filament carbons is found on the surface of Ni-La/SiO₂ supported with SiO₂ support with a lower particle size catalyst suggesting that the catalyst is very active for catalytic activity. Catalyst dispersion for Ni-La/SiO₂ supported with SiO₂ support with a lower particle size is higher than the one with a support of larger particle size, suggesting that catalyst supported on a support of high surface area has a better catalyst dispersion. Higher support loading also gave better catalyst dispersion. In overall, all three objectives of the project have been complied and the work has successfully produced Ni catalyst modified with La supported with SiO₂ with a decent stability and catalytic activity for methane cracking process.

ACHIEVEMENT

- i) Name of articles/ manuscripts/ books published
 - Mohamad Muzakkir Tajuddin, Asmida Ideris, Mazni Ismail (2019) In Situ Glycine–Nitrate Combustion Synthesis of Ni–La/SiO₂ Catalyst for Methane Cracking, *Industrial & Engineering Chemistry Research* 58, 2, 531–538
 - b. M.M. Tajuddin, M.H. Patulla, A. Ideris and M. Ismail (2017) Self-combustion synthesis of Ni catalysts modified with La and Ce using Glycine–Nitrate Process (GNP) *Malaysian Journal of Catalysis* 2, 8–11
- ii) Title of Paper presentations (international/ local)
 - Self-combustion Synthesis of Ni Catalysts Modified with La and Ce Using Glycine-Nitrate Process (GNP) International Conference on Catalysis 2016 (iCAT 2016) Grand Bluewave, Johor Bahru, Malaysia 20- 21st September 2016
- iii) Human Capital Development
 - Muhammad Muzakkir Tajuddin (MSc) MKG15001 Synthesis And Characterization Of Ni-Based Catalyst Modified With Rare Earth Metal Oxides (CeO₂, La₂O₃) For Hydrogen Production
 - b. Muhammad Hasbullah Bin Patulla (Undergraduate Reseach Project)

KA13159 Self-Combustion Synthesis Of Lanthanum-Modified Nickel Catalyst For Hydrogen Production Finished: Sem II 15-16

- Nur Suhana Aisha Isa (Undergraduate Reseach Project) KA13159 Self-Combustion Synthesis of Ceria-Modified Ni Catalyst for Hydrogen Production KA13108 Finished: Sem I 16-17
- d. Mudakarran Murugaiah (Undergraduate Reseach Project) KC13053 Self-Combustion Synthesis of Barium-Modified Nickel for Hydrogen Production KC13053 Finished: Sem I 16-17
- e. Jane Tong Huixia (Undergraduate Reseach Project) KA14071
 In-Situ Preparation of Ni-Based Catalyst Supported On Silica Using Self-Combustion Process Finished: Sem I 17/18
- f. Rais Fakhruddin Bin Roslan (Undergraduate Reseach Project) KC14027 In-Situ Preparation Of Ni-Based Catalyst Supported On Lanthana Using Self-Combustion Process Finished: Sem I 17/18
- g. Teo Jet Yee (Undergraduate Reseach Project) KC14015 In-Situ Preparation Of Ni-Based Catalyst Supported On Alumina Using Self-Combustion Process
 Finished: Sem I 17/18
- iv) Awards/ Others N.A
- v) Others N.A

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APPENDIXES

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