A planar trioctahedral iron(II)–iron(III) hydroxide (green rust, GR) intercalated with dodecanoate (GRC12) has been oxidized by dioxygen to produce the corresponding planar iron(III) (hydr)oxide. The formulae of GRC12 and the final iron(III) product (oxGRC12) were determined to be $\text{Fe}^{\text{II}}_{2.00}\text{Fe}^{\text{III}}_{1.00}(\text{OH})_{5.31}(\text{C}_{12}\text{H}_{23}\text{O}_{2})_{0.66}(\text{SO}_{4})_{0.51}$ and $\text{Fe}^{\text{III}}_{3}\text{O}_{2.18}(\text{OH})_{3.13}(\text{C}_{12}\text{H}_{23}\text{O}_{2})_{0.56}(\text{SO}_{4})_{0.47}$, respectively. oxGRC12 has the same planar layer structure as GRC12, as revealed by identical powder X‐ray diffraction patterns. The electrostatic interactions between the interlayer dodecanoate (C12) anions and the iron hydroxide planar layer were preserved during the oxidation, as shown by FTIR spectroscopy. The high positive charge in the hydroxide layer produced by the oxidation of iron(II) to iron(III) is partially compensated by the deprotonation of hydroxy groups, as shown by X‐ray photoelectron spectroscopy. The Mössbauer quadrupole splitting of the iron(III) doublet increased on oxidation of GRC12, which has been attributed to the distortion of the octahedral sites caused by deprotonation and changes in the coordination site geometry. oxGRC12 is stable in air for more than 2 years. The van der Waals interactions between the alkyl chains of the intercalated dodecanoate anions play a crucial role in stabilizing the structure and hindering the collapse of the iron(II)–iron(III) (hydr)oxide structure during oxidation. This is the first report describing the formation of a stable planar layered octahedral iron(III) (hydr)oxide. oxGRC12 shows promise as a sorbent and host for hydrophobic reagents, and as a possible source of single planar layers of iron(III) (hydr)oxide.

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